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FIELD ANALYTICAL SCREENING PROGRAM: PCP METHOD

INNOVATIVE TECHNOLOGY EVALUATION REPORT

NATIONAL RISK MANAGEMENT RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

NATIONAL EXPOSURE RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
LAS VEGAS, NEVADA 89193

Notice

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Foreword

The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, EPA's research program is providing data and technical' support for solving environmental problems today and building a science knowledge base necessary to manage our ecological resources wisely, understand how pollutants affect our health, and prevent or reduce environmental risks in the future.

The National Risk Management Research Laboratory is the Agency's center for investigation of technological and management approaches for reducing risks from threats to human health and the environment. The focus of the Laboratory's research program is on methods for the prevention and control of pollution to air, land, water and subsurface resources; protection of water quality in public water systems ; remediation of contaminated sites and ground water; and prevention and control of indoor air pollution. The goal of this research effort is to catalyze development and implementation of innovative, cost-effective environmental technologies; develop scientific and engineering information needed by EPA to support regulatory and policy decisions; and provide technical support and information transfer to ensure effective implementation of environmental regulations and strategies.

This publication has been produced as part of the Laboratory's strategic long-term research plan. It is published and made available by EPA's Office of Research and Development to assist the user community and to link researchers with their clients.

E. Timothy Oppelt, Director
National Risk Management Research Laboratory

Abstract

This innovative technology evaluation report (ITER) presents information on the demonstration of the U.S. Environmental Protection Agency (EPA) Region 7 Superfund Field Analytical Screening Program (FASP) method for determining pentachlorophenol (PCP) contamination in soil and water. This method was demonstrated in Morrisville, North Carolina, in August 1993.

The FASP PCP Method was developed by the EPA Superfund Branch for use at Superfund sites. The method uses a gas chromatograph (GC) equipped with a megabore capillary column and either a flame ionization detector (FID) or an electron capture detector (ECD). Gas chromatography is an EPA-approved method for determining PCP concentrations in soil, water, and waste samples. The FASP PCP Method is an abbreviated, modified version of approved methods. Soil and water samples require extraction before GC analysis. To remove interferences caused by petroleum hydrocarbons, an acid-base partition cleanup step is used during the FASP PCP Method.

The FASP PCP Method was found to be field-portable only in a mobile laboratory, must be done in a temperature-controlled environment, and requires a skilled chemist for operation. The detection limit reported by this method for is 0.8 part per million for soil samples and 1 .O part per billion for water samples. PRC used linear regression and inferential statistics to compare the method's data to that from the confirmatory laboratory. When the data sets were evaluated as a whole, the FASP PCP Method did not perform well. However, the demonstration's samples were collected from two different sites, and the method was found to have performed well on samples from the site where petroleum hydrocarbons had not been used as a carrier solvent. This indicates that the problems with the method may have been due to the petroleum hydrocarbons in the soil. The water analysis portion of this demonstration produced similar results.

PRC evaluated field duplicate samples to determine the technology's precision relative to the confirmatory laboratory's. PRC found no significant difference between the precision of the FASP PCP Method and that of the confirmatory laboratory's for soil and water analysis. In addition, no PCP carrier effect on precision was observed.

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List of Abbreviations and Acronyms

Beazer	**Beazer East,** Inc.
CCAL	continuing calibration
DCAA	2,4-dichlorophenylacetic acid
ECD	electron capture detector
EMSL-LV	Environmental Monitoring Systems Laboratory-Las Vegas
EPA	Environmental Protection Agency
ERA	Environmental Resource Associates
ESAT	Environmental Services Assistance Team
FASP	Field Analytical Screening Program
FID	flame ionization detector
GC	gas chromatograph
ICAL	initial calibration
IDW	investigation-derived waste
ITER	Innovative Technology Evaluation Report
Koppers	Koppers Company
μg/kg	micrograms per kilogram
μg/L	microgram per liter
mg/kg	milligram per kilogram
MMTP	Monitoring and Measurement Technologies Program
MtBE	methyl tert-butyl ether
NRMRL	National Risk Management Research Laboratory
ORD	Office of Research and Development
OSWER	Office of Solid Waste and Emergency Response
PCP	pentachlorophenol
PE	performance evaluation
ppb	parts per billion
ppm	parts per million
PRC	PRC Environmental Management, Inc.
QA	quality assurance
QADE	quality assurance and data evaluation
QAPP	Quality Assurance Project Plan
QC	quality control
RCRA	Resource Conservation and Recovery Act
RECAP	Region 7 Environmental Collection and Analysis Program
RPD	relative percent difference
RSD	relative standard deviation
SARA	Superfund Amendments and Reauthorization Act of 1986
SITE	Superfund Innovative Technology Evaluation
SMO	Sample Management Office
SVOC	semivolatile organic compounds
USI	Unit Structures, Inc.

Acknowledgments

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Additional information concerning the demonstration and technology described in this report can be obtained by contacting Mr. Lary Jack, EPA Environmental Monitoring Systems Laboratory, technical project manager, at (702) 798-2373, or Mr. Eric Hess, PRC Environmental Management, Inc., project manager, at (913) 573-1822.

Section 1
Executive Summary

This innovative technology evaluation report (ITER) presents information on the demonstration of the U. S. Environmental Protection Agency (EPA) Region 7 Superftmd Field Analytical Screening Program (FASP) method for determining pentachlorophenol (PCP) contamination in soil and water. This method was demonstrated in Morrisville, North Carolina, in August 1993. The demonstration was conducted by PRC Environmental Management, Inc. (PRC), under contract to the EPA Enviromnental Monitoring Systems Laboratory-Las Vegas (EMSL-LV). The demonstration was developed under the Monitoring and Measurement Technologies Program (MMTP) of the Superfund innovative Technologies Evaluation (SITE) Program.

The FASP PCP Method was demonstrated in conjunction with the demonstrations of four other field screening technologies: (1) the HNU-Hanby Test Kit developed by HNU Systems, (2) the Penta RISc Test Systems developed by EnSys Incorporated, (3) the EnviroGard PCP Test Kit developed by Millipore Corporation, and (4) the Penta RaPID Assays developed by Ohmicron Corporation. The results of these demonstrations are presented in separate reports similar to this one.

The first objective of this demonstration was to evaluate the FASP PCP Method for accuracy and precision in detecting high and low levels of PCP by comparing its results to those from a confirmatory laboratory that used standard EPA-approved analytical methods. These EPA-approved methods are used to provide legally defensible analytical data to monitor or enforce environmental regulations. Because these EPA-approved methods are used by the regulatory community, this demonstration also used these methods. While these methods may include inherent tendencies that may bias data or may include procedures that developers disagree with, they are the best methods for providing legally defensible data as defined by the regulatory community. To remove as much of these inherent tendencies as possible, PRC used post hoc residual analysis to remove data outliers. The FASP PCP technology was also qualitatively evaluated for the length of time required for analysis, ease of use, portability, and operating cost.

The second objective of the demonstration was to evaluate the specificity of the technology. The specificity was evaluated by examining the effects of naturally-occurring matrix effects, site-specific matrix effects, and chemical cross-reactivity. Information on the technology's specificity was gathered from literature, the analysis of demonstration samples, and through a specificity study.

The site selected for demonstrating the technology was the former Koppers Company (Koppers) site in Morrisville, North Carolina. This site was selected because a National Risk Management Research Laboratory (NRMRL) SITE demonstration was planned for this site, allowing a conjunction of logistical and support efforts between NRMRL and EMSL-LV. Another reason for selecting the former Koppers site was that historical documentation indicated that PCP contamination ranged from none detected to 3,200 parts per million (ppm) in soil and from none detected to 1,490 parts per billion (ppb) in groundwater. The PCP carrier used at this site was a mixture of isopropyl ether and butane. Soil and water samples also were collected from the Winona Post site in Winona, Missouri. Samples from the Winona Post site were shipped to the former Koppers site for inclusion as demonstration samples. Winona Post samples were included to broaden the scope of the demonstration by introducing a different sample matrix and a different PCP carrier, diesel fuel.

The FASP PCP Method is designed to provide quick, accurate results for PCP concentrations in soil and water samples. This method also can detect and quantify other phenols. PCP concentrations are reported in either parts per billion or parts per million for soils and parts per billion for waters. This method was developed by the EPA Superftmd Branch for use at Superfund sites.

The FASP PCP Method uses a gas chromatograph (GC) equipped with a megabore capillary column and either a flame ionization detector (FID) or an electron capture detector (ECD) to identify and quantify PCP. Gas chromatography is an EPA-approved method for determining PCP concentrations in soil, water, and waste samples. The FASP PCP Method is an abbreviated, modified version of these methods.

Soil and water samples require extraction before GC analysis. To remove interferences caused by petroleum hydrocarbons, including PCP carriers such as mineral spirits, kerosene, diesel fuel, and fuel oil, an acid-base partition cleanup step. In this step, the method includes petroleum hydrocarbons are removed from the reagent water, while potassium phenates remain in the reagent water. Sample extracts are injected onto a GC, separated with a DB-5 megabore capillary column, and the PCP is identified and quantified using an FID. The sample extracts are then compared to standards to determine whether PCP is present in the sample and, if so, at what concentration. The FASP PCP Method will only provide high parts per billion detection levels of PCP in water when an FID is used. To achieve a lower detection limit, the sample extracts are reanalyzed using an ECD.

The FASP PCP method is field-portable only in a mobile laboratory. It should be used indoors in a temperature-controlled environment. Reagents required for soil and water sample analysis require refrigeration and the GC and extraction fume hood require electricity. The FASP PCP Method requires experienced GC operators to produce reliable results. The average number of demonstration samples extracted, concentrated, and analyzed in one lo-hour day during the demonstration was 14. The detection limit reported by this method for soil samples is 0.8 ppm and 1 .O ppb for water samples.

The FASP PCP Method can be affected by naturally occurring matrix effects such as humic acids, pH, or salinity. Other matrix effects include PCP carriers such as petroleum hydrocarbons or solvents. Due to the nature of chromatography, this method is not greatly influenced by chemical cross-reactivity. The FASP PCP Method was found to be most affected by the diesel fuel used as a PCP carrier solvent. A specificity study performed during the demonstration showed that diesel fuel would provide a positive response when present at a concentration of 10 ppm. Petroleum hydrocarbon interferences were found to affect results for the Winona Post samples.

PRC used linear regression and inferential statistics to compare the technology's data to that from the confirmatory laboratory. When the data sets were evaluated as a whole, a less accurate performance on the Winona Post samples was observed due to the diesel fuel PCP carrier solvent. Both the entire data set and the Winona Post data alone showed that the method produced Level 1 data. However, the method performed well when the samples from the former Koppers site were examined separately. Within this data grouping, the technology produced Level 2 data, which was statistically similar to that from the confirmatory laboratory or that could be mathematically corrected to become similar to that from the confirmatory laboratory. Generally, if 10 to 20 percent of the soil samples (not contaminated with petroleum) are sent to a confirmatory laboratory, then the results from the other 80 to 90 percent can be corrected. This could result in a significant savings in analytical costs. The water analysis portion of this demonstration produced similar results. The FASP PCP Method produced Level 2 data for the samples collected from the former Koppers site. The regression analysis and the Wilcoxon Signed Ranks Test indicated that the technology's data is strongly correlated to the confirmatory data, but is statistically different. This means that the FASP PCP Method's data must be mathematically corrected by having 10 to 20 percent of its samples slated for confirmatory analysis. The Winona Post data showed that even when using sample cleanup, the method produces Level 1 data that is both dissimilar to the confirmatory data and that cannot be mathematically corrected.

PRC evaluated field duplicate samples to determine the technology's precision relative to the confirmatory laboratory's. PRC found no significant difference between the precision of the FASP PCP Method and that of the confirmatory laboratory's for soil and water analysis. In addition, no PCP carrier effect on precision was observed.

Section 2
Introduction

This ITER presents information on the demonstration of the FASP method designed to detect PCP in soil and water. PRC conducted the demonstration under the EPA SITE Program. The FASP technology was demonstrated in conjunction with the demonstrations of four other technologies: (1) the Penta RISc Test System developed by EnSys Incorporated, (2) the EnviroGard PCP Test Kit developed by Millipore Corporation, (3) the Penta RaPID Assay developed by Ohmicron Corporation, and (4) the HNU-Hanby Test Kit developed by HNU Systems. The results of these other technologies are presented in separate reports similar to this one. All tables and figures referenced in this document are presented at the end of their respective sections.

EPA Site Program and MMTP: An Overview

At the time of the. Superfund Amendments and Reauthorization Act of 1986 (SARA), it was well recognized that the environmental cleanup problem needed new and better methods. As a result, the SITE Program was created to fulfill a requirement of SARA that EPA address the potential of alternative or innovative technologies. EPA made this program a joint effort between the Office of Solid Waste and Emergency Response (OSWER) and the Office of Research and Development (ORD). The SITE Program includes four parts:

- The Demonstration Program (for remediation technologies)
- The Emerging Technology Program
- The Monitoring and Measurement Technologies Program (MMTP)
- The Technology Transfer Program

The largest part of the SITE Program is concerned with treatment technologies and is administered by ORD's NRMRL in Cincinnati, Ohio. However, the MMTP component is administered by EMSL-LV. The MMTP is concerned with monitoring and measurement technologies that identify, quantify, or monitor changes in contaminant occurring at hazardous waste sites or that are used to characterize a site.

The MMTP seeks to identify and demonstrate innovative technologies that may provide less expensive, better, faster, or safer means of completing this monitoring or characterization. Managers of hazardous waste sites are often reluctant to use any method, other than conventional ones, to generate critical data on the nature and extent of contamination. In addition, the courts generally recognize data generated by conventional laboratory methods; nevertheless, there is a tremendous need to generate data more cost- effectively. Therefore, the EPA must identify innovative approaches, and through verifiable testing of the technologies under the SITE Program, insure that the innovative technologies are equivalent to or better than conventional technologies.

The Role of Monitoring and Measurement Technologies

Measurement and monitoring technologies are needed to assess the degree of contamination, to determine the effects of contamination on public health and the environment, to supply data for selection of appropriate remedial action, and to monitor the success or failure of selected remedies. Thus, the MMTP is concerned with evaluating screening technologies, including remote sensing, monitoring, and analytical technologies.

Candidate technologies may come from within the federal government or from the private sector. Through the program, developers are allowed to rigorously evaluate the performance of their technologies. By distributing the results and recommendations of those evaluations, the market for the technologies is enhanced.

Defining the Process

The demonstration process begins by canvasing the EPA 10 regional offices (with input by OSWER and ORD) to determine their needs. Concurrently, classes of technologies are identified. An ideal match is made when there is a clear need by EPA regions and a number of technologies that can address that need. Demonstrations are designed to judge each technology against existing standards and not "one against the other."

A demonstration is designed to provide for detailed quality assurance and quality control (QA/QC) to insure that a potential user can evaluate the accuracy, precision, representativeness, completeness, and comparability of data derived from the innovative technology. In addition, a description of the necessary steps and activities associated with operating the innovative technology is prepared. Cost data, critical to any environmental activity, are generated during the demonstration to allow a potential user to make economic comparisons. Finally, information on practical matters such as operator training requirements, detection levels, and ease of operation are reported. Thus, the demonstration report and other informational materials produced by the MMTP provide a real-world comparison of the demonstrated technology to conventional technologies. With cost and performance data, as well as "how to" information, users can determine whether a new technology better meets their needs.

Components of a Demonstration

Once a decision has been made to demonstrate technologies to meet a particular EPA need, the MMTP performs a number of activities. First, MMTP identifies potential participants and determines whether they are interested in participating. Each developer is advised of the general nature of the demonstration and is provided with information common to all MMTP demonstrations. Information is sought from each developer about its technology to insure that the technology meets the parameters of the demonstration. After evaluating the information, MMTP informs all respondents whether or not they have been accepted into the demonstration. While participants are being identified, potential sites also are identified, and basic site information is obtained.

The next component, probably the most important, is the development of plans that describe how the demonstration will be conducted. A major part of the EPA's responsibility is to develop a demonstration plan, a quality assurance project plan (QAPP), and a health and safety plan. While the EPA pays for and has the primary responsibility for these plans, each is developed with input from all of the demonstration's participants. The plans define how activities will be conducted and how the technologies will be evaluated. MMTP also provides each developer with site information and often provides predemonstration samples so the developer can maximize the field performance of its innovative technology. Generally, the developers train EPA-designated personnel to operate the technologies so that performance is not based on the special expertise of the developers. This approach also insures that potential users have valid information on training requirements and the types of operators who typically use a technology successfully.

The field demonstration itself is the shortest part of the process. During the field demonstration, data is obtained on cost, technical effectiveness (compared to standard methods), and limiting factors. In addition, standardized field methods are developed, and daily logs of activities and observations (including photographs or videotapes) are produced. EPA is also responsible for the comparative, conventional method analytical costs and the disposal of any wastes generated by the field demonstration.

The final component of an MMTP demonstration consists of reporting the results and insuring distribution of demonstration information. The primary product of the demonstration is an ITER, like this one, which is peer-reviewed and distributed as part of the technology transfer responsibility of the MMTP. The ITER fully documents the procedures used during the field demonstration, QA/QC results, the field demonstration's results, and its conclusions. A separate QA/QC data package is also made available for those interested in evaluating the demonstration in greater depth. Two-page "Technical Briefs" are prepared to summarize the demonstration results and to insure rapid and wide distribution of the information.

Each developer is responsible for providing the equipment or technology product to be demonstrated, its own mobilization costs, and the training of EPA-designated operators. The MMTP does not provide any funds to developers for costs associated with preparation of equipment for demonstration or for development, and it does not cover the costs developers incur to demonstrate their products.

Rationale for this Demonstration

PCP is a regulated chemical, is included in the EPA Extremely Hazardous Substances List, and is reported in the EPA Toxic Substances Control Act. Recently, PCP regulations under the Resource Conservation and

Recovery Act (RCRA) have been created specifically for wood treatment facilities. PCP is included as a target compound of many EPA-approved analytical methods including: EPA 500 Series Methods 515.1 and 525, EPA 600 Series Methods 604 and 625, and EPA SW-846 Manual Methods 8040, 8151, 8250, and 8270. All these methods use solvent extraction and gas chromatography. Detection and quantitation is performed with FIDs, ECPs, or mass spectrometer detectors. Analyzing samples for PCP using these methods is typically costly and time consuming. EMSL-LV identified the need for effective, accurate, low cost screening technologies that could provide near real-time analytical data for PCP to Superfund and RCRA decisionmakers.

Demonstration Purpose, Goals, and Objectives

The FASP PCP Method was evaluated on its accuracy and precision in detecting high and low levels of PCP in environmental samples, and on the effects, if any, of both PCP carrier and natural matrix interferences. The accuracy and precision of the method was statistically compared to the accuracy and precision of a conventional confirmatory laboratory that used EPA-approved analytical methods. This comparison was also used to determine the highest data quality level that the technology could attain in field applications. For the purpose of this demonstration, three primary data quality levels were defined as follows (EPA 1990):

Level 1: Level 1 data is not necessarily analyte-specific. Technologies that generate Level 1 data provide only an indication of contamination. Generally, the use of these technologies requires sample documentation, instrument calibration, and performance checks of equipment.

Level 2: Level 2 data is analyte-specific. To provide an accuracy check, verification analysis by an EPA-approved method is necessary for at least 10 percent of the samples. The method's analytical error is also quantified. Use of QC procedures such as sample documentation, chain-of-custody procedures, sample holding time criteria, initial and continuing instrument calibration, method blank analysis, rinsate blank analysis, and trip blank analysis is recommended.

Level 3: Level 3 data is considered formal or confirmatory analysis. These data are analyte-specific and generally involve second-method confirmation on 100 ˙ percent of critical samples. Analytical error is quantified (including precision, accuracy, and coefficient of variation) and monitored. The following QC measures are used: sample documentation, chain of custody, sample holding time criteria, initial and continuing instrument calibration, rinsate blank analysis, trip blank analysis, and performance evaluation samples. Detection limits are determined and monitored.

Inherent in this concept of data quality levels is accuracy. Although PRC could not find a reference that defined the expected and quantified accuracy of each data quality level, it imposed common accuracy criteria in defining these data quality levels. Data quality Level 3 is considered the most accurate and is based on a formal analysis by approved methods. Data quality Level 2 is less accurate, but it still does quantify compound concentrations. Data quality Level 1 is the least accurate and is often considered survey data, useful only in identifying the presence or absence of a compound or class of compounds. Because no existing quantification of the criteria defining these data quality levels was found, PRC set the criteria used in this ITER based on the experience of the EPA and PRC personnel involved and on a general survey of environmental consultants who use data.

The FASP PCP Method was also qualitatively evaluated for specificity, the length of time required for its analysis, ease of use, portability, and operating cost.

Section 3
Predemonstration Activities

Several activities were conducted by EMSLLV, PRC, and other demonstration participants before the demonstration began. These activities included identifying developers, selecting demonstration sites, selecting the confirmatory laboratory and analytical methods, conducting predemonstration sampling, and training technology operators. Predemonstration sampling and analysis are normally used to allow developers to refine their technologies and revise their operating instructions, if necessary, prior to the demonstration.

Identifying Developers

EMSL-LV asked that PRC search for technologies that could be included in this demonstration. Based on PRC's search, the FASP method was included.

The Sites and Their Principal Contaminants

To evaluate the field screening technology under field conditions, hazardous waste sites suitable for the demonstration were needed. The following criteria were used to select appropriate sites:

- The technology needed to be demonstrated at sites with a wide range of PCP contamination.
- PCP concentrations at the sites had *to* be well characterized and documented.
- The sites had to be accessible for conducting demonstration activities without interfering with other activities being conducted on site.
- Because various carriers have been used with PCP and because those carriers may influence the technology, it was determined that the sites used should offer two different carriers.

The former Koppers wood treatment site was selected as one of the two sites for this demonstration based on these criteria. This site also was selected because EPA's NRMRL was planning a SITE demonstration of the ETG Environmental, Inc., Base-Catalyzed Decomposition technology at the site, and choosing the former Koppers site would allow for a conjunction of logistical and support efforts between NRMRL and EMSL-LV. The second demonstration site selected was the Winona Post site wood treatment facility. The Winona Post site is contaminated with PCP in a diesel fuel carrier solvent. The former Koppers site is contaminated with PCP in butane and isopropyl ether carrier solvents.

The former Koppers site is located in Morrisville, North Carolina, at the intersection of Highway 54 and Koppers Road. The site is currently owned by two companies : Beazer East, Inc. (Beazer), and Unit Structures, Inc. (USI). The portion of the site owned by Beazer is inactive. The portion of the site owned by US1 is currently used as a wood laminating facility. The site occupies about 52 acres and includes the wood laminating building, an office, and several warehouses. Surrounding land use is a mixture of commercial, light industrial, and rural residential. During previous investigations at the former Koppers facility, samples were collected from the following media: soil, groundwater, surface water, sediment, and fish. Sampling revealed PCP concentrations ranging from not detected to 3,200 ppm in soils and from not detected to 1,490 ppb in water.

The Winona Post site is located in Winona, Missouri, on Old Highway 60 West. It has operated as a sawmilling and wood preserving facility since at least the early 1950s. The sawmilling, wood preserving, and storage areas of the facility cover about 4 acres. The remaining portion of the 40-acre facility is wooded and largely undeveloped. The main features of the facility include a sawmill, office, treatment building, debarker, storage building, and pond. Currently, the company uses a premixed solution of 5 percent PCP in diesel fuel. The solution is stored in a 20,000-gallon aboveground storage tank located adjacent to the treatment building. In the past, the Winona Post Company mixed its own solution from concentrated PCP. Prior to the mid-1950s. the Winona Post Company treated wood with cresol. In 1992, six samples collected at the Winona Post site revealed PCP concentrations ranging from 886 to 24,000 ppm in soil and sediment samples and

from 10 to 528 ppm in surface water samples.

PCP is an organic chemical with an empirical formula of C_6Cl_5OH and a molecular weight of 266 grams per mole. PCP has a melting point of 191 °C and a boiling point of 310 "C. The specific gravity of PCP is 1.978 grams per cubic centimeter. PCP is described as almost insoluble in water, with 8 milligrams able to dissolve into 100 milliliters of water. The octanol ratio coefficient of PCP is 6,400, which indicates that PCP is tightly bound to the soil matrix when it is released into the environment. PCP is used as a wood preservative, an insecticide, a preharvest defoliant, a slimicide, and a defoaming agent. The largest user of PCP is the wood treating industry. For treating wood, PCP is usually diluted to a 5 percent solution with solvents such as mineral spirits, kerosene, diesel fuel, or fuel oil. PCP also has been applied to wood with methylene chloride and liquified petroleum gas, such as butane. It has been manufactured under numerous trade names.

Selecting the Confirmatory Laboratory and Analytical Methods

Before this demonstration, the EPA Region 7 Laboratory arranged for all soil samples to be analyzed under the Region 7 Environmental Collection and Analysis Program (RECAP) Contract and all water samples to be analyzed under its Environmental Services Assistance Team (ESAT) Contract. SW-846 protocols for Level 3 data were to be used to analyze soil and water samples during this demonstration. All samples were to be extracted by EPA Method 354OA and analyzed by EPA Method 8270A. Any soil samples in which PCP was not detected using Method 8270A were to be reanalyzed by Method 8151A calibrated to PCP. Any groundwater samples in which PCP was not detected using Method 8270A were to be reanalyzed using Method 515.1. All of these analytical methods are well established and approved by EPA. QA procedures, reporting requirements, and data quality objectives of these methods are consistent with the goals of the SITE Program.

Training Technology Operators

Analysis with the FASP PCP Method was conducted by a PRC operator. Before the demonstration began, this individual was trained in how to use the method. The training involved a review of operating procedures and instruction provided by the lead chemist.

Predemonstration Sampling and Analysis

In July 1993, PRC prepared a predemonstration sampling plan (PRC 1993a), and on July 12, 1993, PRC collected predemonstration soil samples from areas at the former Koppers facility previously identified as containing high, medium, low, and not detected concentrations of PCP. PRC analyzed one replicate of each sample using the FASP PCP Method. Predemonstration samples did not exhibit their expected PCP concentrations. These samples were not analyzed by a confirmatory laboratory because the contracts for the confirmatory analyses were not yet finalized. No predemonstration samples were collected from Winona Post because this site had not been added to the demonstration plan at the time of the predemonstration sampling.

Section 4
Demonstration Design and Description

This section describes the organization of the demonstration, presents an overview of the demonstration's design, and details all deviations from the developer- and EPA-approved, demonstration plan. Among the key portions of the demonstration plan presented here are the types of data collected and the statistical methods used to determine the accuracy and precision of the technology. A detailed description of the demonstration is presented in the demonstration plan (PRC 1993b).

Demonstration Design

The primary objective of the demonstration was to evaluate the FASP PCP Method for its effectiveness in detecting PCP in soil and water when operated in field conditions. This objective included defining the precision, accuracy, cost, and range of usefulness for the technology. A secondary objective was to define data quality objectives that the technology can be used to address. The evaluation was designed so that results from the technology could be compared to those of a confiiatory laboratory that analyzed each sample using standard EPA-approved methods. The design limited, as much as possible, those elements of sample collection and analysis that would interfere with direct comparison of the results. These elements included heterogeneity of the samples and interference from other chemicals or other controllable sources.

The design also insured that the data was collected in a normal field environment. To achieve this, the method was used in a trailer located at the former Koppers site. The operator was trained by the PRC lead chemist. However, the operator obtained all results on his own and reported the results once he believed the results were accurate and precise. Standard QC samples were analyzed with each batch of environmental samples. Numerous laboratory and field duplicate samples were included among those analyzed to insure a proper measure of precision. The technology was also tested for common interferants. Qualitative measures, such as portability and ease of operation, were noted by the operator.

Overall, the demonstration was executed as described in the demonstration plan (PRC 1993b), which included the QAPP. The final version of the plan was approved by all participants and developers before the demonstration began.

Implementation of the Demonstration Plan

For the demonstration, PRC collected 98 soil samples, 14 soil sample field duplicates, 10 water samples, and 6 water sample field duplicates. Each soil sample was thoroughly homogenized and then split into replicate samples. One replicate from each sample was submitted to the confirmatory laboratory; another replicates was analyzed in a trailer at the former Koppers site using the FASP PCP Method. In addition to these samples, two soil performance evaluation (PE) samples and three water PE samples were analyzed.

The final demonstration plan called for the collection of 90 soil samples with the following distribution: (1) 40 samples containing 0 to 100 ppm PCP; (2) 25 samples containing 100 to 1,000 ppm PCP; and (3) 25 samples containing greater than 1,000 ppm PCP. During this demonstration, 98 soil samples were collected. The actual distribution of these samples, when the demonstration was complete, was as follows: (1) 60 samples contained 0 to 100 ppm PCP; (2) 16 samples contained 100 to 1,000 ppm PCP; and (3) 22 samples contained greater than 1,000 ppm PCP. This skewing of the sample set to the 0 to 100 ppm range should not affect the usability of this report, because the majority of EPA PCP soil action levels occur in the 20 to 100 ppm range.

Of the samples collected for the demonstration, 53 soil samples, 9 soil field duplicates, 5 water samples, and 5 water field duplicate samples were collected at the former Koppers site. Soil samples were collected from

areas known to exhibit a wide range of PCP concentrations. The areas sampled ranged in PCP concentration from not detected to 3,220 ppm. Most of the samples were collected from areas characterized during the remedial investigation. Water samples were collected from five existing grotmdwater monitoring wells located on the former Koppers site. The PCP concentrations in these wells were well documented from past sampling. The PCP concentrations sampled ranged from not detected to nearly 1,500 ppb.

Of the samples collected for the demonstration, 45 soil samples, 5 soil field duplicates, 5 water samples, and 1 water duplicate sample were collected at the Winona Post site. Soil samples were collected in areas believed to be contaminated with high (greater than 1,000 ppm), medium (100 to 999 ppm), and low (less than 99 ppm) concentrations of PCP. The identification of these areas was based on past sampling data and visual signs of waste disposal. Water samples were collected from the only surface water located on or near the site. All of the Winona Post samples were collected, packaged, and shipped to the former Koppers site using methods detailed in the demonstration plan (PRC 1993b).

Field Modifications to the Demonstration Plan

Two field modifications were made to the approved demonstration plan. First, fluorescein was not added to soil samples from the former Koppers site. The nature of the soil samples at both the former Koppers site and the Winona Post site allowed easy and thorough homogenization. The saturated stiff clay matrix for which the fluorescein additions were designed was not encountered at the former Koppers site and thus, for consistency, this technique was eliminated at both sites. PRC believes that the elimination of the fluorescein from the homogenization process was offset by the long homogenization times used during this demonstration. To further examine this approach, PRC conducted a side-by-side comparison of homogenization with and without fluorescein. Samples from the former Koppers site were used for this comparison. The dry nature of the soil required that it be hydrated with water to allow visible distribution of the fluorescein. The addition of water and fluorescein caused a two-unit increase in the soil sample pH This alteration of the sample chemistry coupled with the reactive nature of PCP invalidated the fluorescein homogenization approach for environmental applications. PRC used an EPA-approved homogenization method and applied it to each sample for 10 to 15 minutes. This method involved vigorous kneading of the sample in a clear plastic bag.

The second modification to the approved demonstration plan involved sampling the water matrix. This change was made because the EPA Region 7 project sponsor altered the design of the demonstration with regard to the evaluation of the water assays. The EPA sponsor required that the number of water samples collected and analyzed for the demonstration be reduced to a total of 5 to 10 samples. The approved demonstration plan called for the collection of 50 groundwater samples. To maximize the usefulness of the reduced number of water samples, PRC and EMSL-LV agreed to combine data from both sites if they could be shown statistically to come from the same distribution, thereby increasing the sample set size. Also, the EPA Region 7 sponsor agreed to allow the following: (1) the Region 7 Laboratory would split and analyze samples from Winona Post in sample-plus duplicate (split) pairs; (2) excess water from the original Winona Post water sample that was duplicated would be used for laboratory QA/QC; and (3) only five monitoring wells would be sampled at the former Koppers site, and each sample would be duplicated. This approach would have resulted in five paired samples (the sample plus its duplicate) from each site. This in turn would have provided five samples from each site for an accuracy assessment, and five paired samples from each site for a precision assessment. Although these are minimal sample sizes, this design was believed to provide the most useful data given the reduction in analytical resources that the EPA Region 7 sponsor required. However, when PRC delivered the soil and water samples from the former Koppers site, it learned that the Winona Post water samples had been extracted and analyzed as they were delivered, that is, as five environmental samples with one duplicate. This failure to follow the modified experimental design resulted in only four single sample results and one duplicate result for precision analysis for this data set. The Koppers data set consisted of five duplicate pairs.

Data Collection

The technology operator prepared a subjective evaluation of how difficult the technology was to use. Other qualitative measures assessed included portability, ruggedness, instrument reliability, and health and safety considerations. Information on these qualitative factors was collected both by the operator of the technology and by the lead chemist. To evaluate accuracy and precision, all samples collected for analysis were split between the technology and the confirmatory laboratory. Statistical methods used to compare the results of the two methods are detailed below. The cost of using this technology was also assessed. Cost, for the purposes of this demonstration, included expendable supplies, nonexpendable equipment, labor, and investigation-

Instrument reliability was evaluated by monitoring the equipment's ability to maintain its calibration. An initial calibration (ICAL) was performed at the start of the demonstration. The instrument calibration was monitored daily through continuing calibrations (CCAL) using the mid-level concentration of the PCP standards. During the CCAL the peak response obtained is compared to the average peak response obtained during the ICAL. These responses are compared by determining the RPD between responses. An RPD value of less than or equal to 25 percent is required to continue sample analysis. If the RPD is higher than 25 percent, another ICAL must be performed. Over the 13 days of the demonstration, three ICALs and 13 CCALs were performed. Out of three ICALs, two were performed for soil analysis, and one was performed for water analysis. Eleven CCALs were performed for the analysis of soil samples, and two CCALs were performed for the analysis of water samples. CCALs were performed on each day of sample analysis. On the eleventh day of the demonstration, the CCAL for soil analysis was out of control as a result of analyzing soil samples from the Winona Post site, which contained large amounts of petroleum hydrocarbons. Due to the heavy contamination with petroleum hydrocarbons in the samples and extracts, the septa and the glass liner in the injection port became contaminated, and the column became saturated with hydrocarbons. After determining the problem, the septa and the glass liner were replaced, and the column was conditioned at elevated oven temperature to desorb the petroleum hydrocarbons. Another ICAL was performed after column conditioning; although the relative standard deviation (RSD) for PCP was 33 percent in the second ICAL, this RSD was deemed acceptable, and the average calibration factor was used for sample quantitation. The two CCALS performed after the second ICAL were within the new control limits.

A new ICAL was performed for the analysis of water samples. The CCAL performed for water analysis was within control limits; however, the CCAL performed at the end of the analysis did not fall within control limits. The retention time of the specific PCP peak also was monitored through the CCALs. The retention time of this peak shifted throughout the demonstration. To maintain an accurate retention time window for this peak, the window was adjusted.

Another reliability factor evaluated was the consistency of the data system to properly draw a baseline for standards and samples. Baselines were drawn consistently for samples from the former Koppers site. However, samples from Winona Post site were highly contaminated with petroleum hydrocarbons, causing the baseline to drift.

Operators of the method need to be aware of hazards due to the chemicals used, particularly methyl tert-butyl ether (MtBE), sodium sulfate, and sulfuric acid. MtBE is used to extract PCP from samples and to dilute standard solutions. MtBE is an explosive and flammable solvent and should be handled only under a flame hood. Care should be taken when using MtBE to avoid inhalation and direct contact with the liquid and fumes. Ignition sources such as open flames should also be avoided, and a dry chemical fire extinguisher should be available in case of fire. Sodium sulfate is used to chemically dewater soil samples. Sodium sulfate is a fine granular powder and can be a skin and eye irritant. Protective clothing and safety glasses should be worn when using sodium sulfate. Sulfuric acid is used to reduce the pH of the samples. Sulfuric acid can cause chemical burns when spilled on the skin. Chemical-resistant clothing, gloves, and safety glasses are required for protection from sulfuric acid. An adequate source of water should be available in case the acid comes in contact with the skin. Eyewash solutions also should be available.

This method uses various types of glassware to analyze samples. Using glass always presents a possibility of breakage and injury. Glassware should be handled in a safe, careful manner. Broken glassware should not be used and should be disposed of in a safe manner. The ECD contains Nickel-63, a radioactive material. The amount of radioactive material in the ECD is minimal, and it is stored in a sealed container. Nevertheless, the container must be checked for possible leakage twice a year. Leakage of the radioactive material above federally regulated limits requires immediate attention. The FID uses hydrogen gas, which is explosive and stored under high pressure. Care must be taken to secure the hydrogen gas cylinders to avoid cylinder damage.

The total cost of the analytical equipment used during this demonstration was $23,214. This cost includes the GC equipped with an ECD and FID, the autosampler, the data system, all equipment required to set up the GC, and installation. Costs per component are: $13,149 for the GC; $3,865 for the autosampler; and $6,200 for the data system. Analytical equipment can also be rented from a number of companies. A general survey of current market costs for comparable equipment indicates ranges from $1,500 to $2,500 per month. Most rental companies require a minimum rental time period, but this may be negotiable. Rental rates usually include delivery and setup but do not include GC columns. Many companies also offer rent-to-own or lease-to-own options for analytical equipment. The cost of reagents and equipment needed to perform the extraction, preparation, and analysis of soil samples

derived waste (IDW) disposal. These costs were tracked during the demonstration.

Statistical Analysis of Results

For both the innovative technology and the confirmatory laboratory, two data sets were created: one for soil samples and the other for water samples. In addition, each water and soil data set was composed of two subsets, one for the samples taken from the former Koppers site and one for those collected at the Winona Post site. This grouping was intended to assess potential PCP carrier effects. A third data separation involved grouping the site-specific data sets into results greater than 100 ppm PCP and less than 100 ppm PCP. This grouping was intended to assess potential concentration effects on the data analysis.

These data sets were prepared for the statistical analysis following methods detailed in the approved demonstration plan. When comparing duplicate samples or when comparing the results of a technology to those from the confirmatory laboratory, sample pairs that contained a nondetect were removed from the data sets. While other statistical methods can be used when nondetects are encountered, PRC believed that the variance introduced by eliminating these data pairs would be less than, or no more than equal to, the variance produced by giving not detected results an arbitrary value.

lntramethod Comparisons

Sample results from the technology were compared to their duplicate sample results and to other QA/QC sample results. These comparisons are called intramethod comparisons. Intramethod accuracy was measured by assessing each technology's performance in analyzing PE samples. If the method produced a result considered accurate by the company that produced the PE samples, the technology was considered to have acceptable intramethod accuracy for this demonstration. Intramethod precision was assessed through the statistical analysis of relative percent differences (RPD). First, RPDs of the results for each sample pair, in which both the sample and its duplicate were found to contain PCP, were determined. RPDs then were compared to upper and lower control limits. When using conventional technologies, such data is often available from analysis of samples collected during previous investigations. Because the technology being demonstrated was itself being assessed, the control limits used were calculated from data provided during this investigation. To determine these control limits, the standard deviation of the RPDs was calculated for each technology. This standard deviation was then multiplied by two and added to its respective mean RPDs. This established the upper control limit for the technology. Because an RPD of zero would mean that the duplicate samples matched their respective samples perfectly, zero was used as the lower control limit. This resulted in a large range of acceptable values. Because duplicate analyses seldom match perfectly, even for established technologies, all samples that fell within the control limits were considered acceptable. PRC determined that if at least 90 percent of the duplicate samples fell within these control limits, the technology had acceptable intramethod precision.

Intermethod Comparisons

Data sets from the FASP PCP Method also were statistically compared to results from the confirmatory laboratory, and the precision of the method was statistically compared to the precision of the confirmatory laboratory. These comparisons are called intermethod comparisons. In both cases, results from the confiiatory laboratory were considered to be as accurate and precise as analytically possible. Statistical methods used to determine intermethod accuracy included linear regression analysis and the Wilcoxon Signed Ranks Test. Before undertaking the regression analysis, PRC further prepared the data sets by averaging the field duplicate results. This approach ensured that samples were not unduly weighted. PRC calculated linear regression by the method of least squares. Calculating linear regression in this way makes it possible to determine whether two sets of data are reasonably related, and if so, how closely. Calculating linear regression produces an equation that can be visually expressed as a line. Three factors are determined during calculations of linear regression: (1) the y-intercept, (2) the slope of the line, and (3) the correlation coefficient, also called an r^2. All three of these factors must have acceptable values before a technology's accuracy was considered to meet Level 3 data quality requirements.

The r^2 expresses the mathematical relationship between two data sets. If the r^2 is one, then the two data sets are directly related. Lower r^2 values indicate less of a relationship. Because of the heterogeneous nature of environmental samples, r^2 values between 0.85 and 1 were considered to meet data quality Level 3 accuracy requirements; r^2 values between 0.75 and 0.85 were considered to meet data quality Level 2 accuracy requirements; and r^2 values below 0.75 were considered not accurate, meeting only Level 1 accuracy requirements at best.

If the regression analysis resulted in an r^2 between 0.85 and 1, then the regression line's y-intercept and

slope were examined to determine how closely the two data sets matched. A slope of one and a y-intercept of zero would mean that the results of the technology matched those of the confirmatory laboratory perfectly. Theoretically, the farther the slope and y-intercept differ from these expected values, the less accurate the technology. Nevertheless, a slope or y-intercept can differ slightly from their expected values without that difference being statistically significant. To determine whether such differences were statistically significant, PRC used the normal deviate test statistic. This test statistic results in a value that is compared to a table. The value at the 90 percent confidence level was used for the comparison. To meet data quality Level 3 requirements, both the slope and y-intercept had to be statistically the same as their ideal values.

If the r^2 was between 0.75 and 0.85, and one or both of the other two regression parameters were not equal to their ideal, the technology was considered to be inaccurate but producing Level 2 quality data. Results in this case could be mathematically corrected if 10 to 20 percent of the samples were sent to a confiitory laboratory. Analysis of a percentage of the samples by a confiimatory laboratory would provide a basis for determining a correction factor. Only in cases where the r^2, the y-intercept, and the slope were all found to be acceptable did PRC determine that the technology was accurate, meeting Level 3 data quality requirements.

Data placed in the Level 1 category had r^2 values less than 0.75, the data was not statistically similar to the confirmatory data, based on parametric testing, or the results did not meet the manufacturer's performance specifications.

A second statistical method used to assess the intermethod accuracy of the data from each technology was the Wilcoxon Signed Ranks Test. This test is a nonparametric method used to compare matched pairs of data. It can be used to evaluate whether two sets of data are significantly different. The test requires no assumption regarding the population distribution of the two sets of data being evaluated other than that the distributions will occur identically. In other words, when one data point deviates, its respective point in the other set of data will deviate similarly. Because the only deviation expected during the demonstration was a difference in the concentrations reported by each technology, the two sets of data were expected to deviate in the same way. The Wilcoxon Signed Ranks Test calculation uses the number of samples analyzed and a ranking of the difference between the result obtained from the demonstrated technology and the corresponding result from the confirmatory laboratory. The rankings can be compared to predetermined values on a standard Wilcoxon distribution table, which indicates whether, overall, the two methods have produced similar results.

Finally, the precision of the technology was statistically compared to the precision of the confirmatory laboratory using Dunnett's Test. This test was used to assess whether the precision of the technology and that of the confirmatory laboratory were statistically equivalent. First, PRC determined the mean RPD for all samples and their respective duplicates analyzed by the confirmatory laboratory. The RPD of each duplicate pair analyzed by each of the technologies was then statistically compared to this mean. It should be noted that a Dunnett's result showing the precisions are not similar does not mean that the precision of the technology was not acceptable, only that it was different from the precision of the confirmatory laboratory. In particular, the Dunnett's Test has no way of determining whether or not any difference between the two data sets actually resulted because a technology's data was more precise than the confirmatory laboratory's. The Dunnett's results were verified by the Wilcoxon Signed Ranks Test.

Section 5
Confirmatory Analysis Results

All samples collected during this demonstration were submitted to the EPA Region 7 Laboratory for confirmatory analysis. Water samples were analyzed by the EPA Region 7 Laboratory under the ESAT Contract, and soil samples were analyzed under the RECAP contract. The ESAT Contract analysis was conducted at the EPA Region 7 Laboratory. The EPA Region 7 Laboratory forwarded all soil samples to the RECAP Laboratory. Discussion of the analyses at both laboratories is presented below. Analytical data provided by the confirmatory laboratories is shown in Section 6 with that of the FASP PCP Method.

Confirmatory Laboratory Procedures

EPA Region 7 Laboratory Quality Assurance and Data Evaluation (QADE) Branch personnel conducted a Level 2 data review on results provided by the confirmatory laboratories. A Level 2 data review does not evaluate raw data or check calculated sample values. A review of the raw data and a check of the calculations was performed by QC personnel from each of the confirmatory laboratories before submitting the data package to the EPA Region 7 Laboratory QADE Branch. PRC was not able to review all of the raw data generated from the analysis of samples. However, PRC did review the laboratory case narratives and the EPA Region 7 Laboratory QADE Branch comments generated by the Level 2 data review.

The following sections discuss specific procedures used to identify and quantitate semivolatile organic compounds (SVOC), and specifically PCP, using the following methods: SW-846 Method 8270A (soil and water), SW-846 Method 8151A (soil), and EPA Method 515.1 (water).

Sample Holding Times

All of the analytical methods used for confirmatory analysis require that all sample extractions be completed within 7 days from the time a sample is collected. Due to the stability of PCP, EPA's ORD Methods Validation Section extended these holding time requirements by 4 days for this demonstration. All sample extracts must be analyzed within 40 days of validated sample receipt. All holding time requirements were met for samples collected during this demonstration.

Sample Extraction

EPA Method 3550 was used to extract soil samples prior to analysis by EPA Method 8270A. This method involves sonication extraction of the soil using methylene chloride. The confirmatory laboratory used both the low concentration extraction method and the high concentration extraction method discussed in EPA Method 3550. To determine the appropriate extraction method to use for the analysis of individual soil samples, the confirmatory laboratory screened each sample using the screening techniques recommended in EPA Method 8270A. EPA Method 3510A was used to extract water samples and involves a separatory extraction of the water with methylene chloride. To ensure that phenolic compounds, such as PCP, were adequately extracted from the water samples, two extractions of each water sample were performed. The pH of the water was adjusted to greater than 12 and extracted, then the pH of the water sample was adjusted to below 2 and extracted. The two sample extracts were then combined for sample analysis.

Low-level detection analytical methods for PCP included different procedures for sample extraction. The method used for the soil samples, EPA Method 8151A, involves an acidification of the soil sample, followed by an ultrasonic extraction with methylene chloride. This extraction is similar to the EPA Method 3550 sonication extraction. The soil sample extract was then taken through an acid-base partition to remove potentially interfering compounds from the sample extract. The sample extract was then concentrated and taken through

a diazomethane derivatization. This procedure replaces the hydrogen atom of the alcohol group with a methyl anion. This derivatization removes the polarity associated with PCP and enables improved chromatographic behavior. PCP standards used for sample identification and quantitation were taken through the same derivatization steps as samples to allow a direct comparison of concentration. That is, no correction factor needs to be used for the molecular weight of the derivatization product.

The low level detection analytical method used for water samples, EPA Method 515.1, involves a separatory extraction of the water sample with methylene chloride. The pH of the water samples was adjusted in a similar manner to the pH adjustment used for the water samples extracted with EPA Method 3510A. In EPA Method 515.1, the solvent extract from the basic extraction is discarded because it contains no PCP. This step also removes potential interferences. The water sample extract is then concentrated and derivatized in the same manner as the soil sample extracts. Again, this derivatization removes the polarity associated with PCP and provides improved chromatographic behavior. PCP standards used for sample identification and quantitation were taken through the same derivatization steps as the samples to allow a direct comparison of concentration.

Detection Limits and Initial and Continuing Calibrations

The detection limit for soil samples analyzed by EPA Method 8270A was reported as 0.330 ppm. The detection limit for soil samples analyzed by EPA Method 8151A was reported as 0.076 ppm. The reported detection limit for water samples analyzed by EPA Method 8270A was 50 ppb. The detection limit for water samples analyzed by EPA Method 515.1 was reported as 0.076 ppm. Method-required initial and continuing calibration procedures were appropriately conducted, and all method-required criteria for these calibrations were met.

Sample Analysis

The confirmatory laboratories performed sample analysis by first analyzing samples using EPA Method 8270A. Based upon the screening results, the samples were extracted with either the low concentration method or the high concentration method found in EPA Method 3550. Samples that did not provide a positive response for PCP with EPA Method 8270A were analyzed by one of two low-level detection methods: EPA Method 8151A for soil samples and EPA Method 515.1 for water samples.

For EPA Method 8270A, compound identification was required to meet two criteria: (1) the sample component relative retention time was to fall within ± 0.06 relative retention time units of the standard component, and (2) the mass spectrum of the sample compound was to correspond with the standard compound mass spectrum.

Soil and water samples found to contain no PCP during the EPA Method 8270A analysis were analyzed using EPA Methods 8151A and 515.1, respectively. The presense of PCP was identified if a sample peak eluted within the retention time window established during the initial calibration.

Quality Control Procedures

Method blanks are used to monitor the presence of laboratory-induced contamination. The EPA Region 7 Sample Management Office (SMO) provided blank soil and blank water samples for use as method blank samples during the analysis of demonstration samples. An acceptable method blank must not provide a positive response for the target compounds above the reported detection limit. Method blank samples were stored, extracted, and analyzed in exactly the same manner as the demonstration samples. Results for all method blank samples extracted and analyzed along with the demonstration were found to be acceptable.

Internal standards were used to analyze demonstration samples by EPA Method 8270A. Internal standards were added to all standards, blanks, samples, and QC samples prior to injection into the analytical system. The internal standards were used to provide response factors for each of the target compounds. During the analysis of soil samples, seven samples exhibited internal standard responses that were outside of the QC limits of 50 to 150 percent recovery. All of the affected samples provided internal standard responses that were less than 50 percent. The soil samples affected were samples 038, 060, 062, 068, 090, 091, and 095. Three of these samples (090, 091, and 095) were found to contain no detectable levels of PCP, and no corrective action was taken. Instead, they were reanalyzed using EPA Method 8151A. The remaining samples were reanalyzed to verify that the internal standard response was below 50 percent recovery. The reanalysis showed that internal standard response was below 50 percent recovery. No corrective action was taken by the laboratory, which attributed the low recovery to matrix effects inherent to the samples. In the Region 7 Laboratory QADE Branch review of the data, the same conclusion was reached.

Surrogate standards were used to evaluate the efficiency of the extraction and analysis processes and to evaluate matrix affects. Surrogate standards used for EPA Method 8270A include deuterated standards that provide a different mass spectrum when compared to the nondeuterated compound. All surrogate standard recoveries for soil samples fell within the acceptance ranges. The data review performed by Region 7 QADE indicated that surrogate recoveries for some of the water samples were outside of the acceptance ranges, but no information indicated which samples or how many samples fell outside surrogate recovery acceptance ranges. Corrective action was not taken because the acceptance ranges listed in the method are for advisory purposes only. The surrogate standard used for EPA Methods 8151A and 515.1 was 2,4 dichlorophenylacetic acid (DCAA). The acceptance range for DCAA was determined by the RECAP and Region 7 Laboratories through a statistical analysis of 30 or more standard surrogate recoveries. The mean and standard deviation were then calculated, and the acceptance range was determined by applying a ± 3 standard deviations around the mean. All samples analyzed by EPA Methods 8151A and 515.1 provided surrogate recoveries that fell within the laboratory-generated control limits.

Matrix spike samples consisted of aliquots of original sample with a known concentration of the target compounds added. The EPA Region 7 Laboratory SMO designated the samples to be used as matrix spike samples. Designated soil samples included samples 036, 048, 053, 073, 087, and 098, all analyzed using EPA Method 8270A. and sample 089, analyzed using EPA Method 8151A. Designated water samples included samples 101 and 111, analyzed using EPA Method 515.1, and sample 104, analyzed using EPA Method 8270A. Soil matrix spike samples analyzed using EPA Method 8270A were spiked with all the target compounds reported by the method. Water sample matrix spike samples analyzed using EPA Method 8270A were spiked with nine of the target compounds reported by the method. Matrix spike samples analyzed with EPA Methods 8151A and 515.1 were spiked with only PCP. Soil matrix spike data for PCP is shown in Table 5-1, and water matrix spike data for PCP is shown in Table 5-2.

Soil sample matrix spike recoveries were greatly influenced by the high concentrations of PCP present in the original sample relative to the amount spiked. Only sample 098 resulted in recoveries for both the matrix spike and matrix spike duplicate sample that could be considered acceptable. A clear evaluation of the effects of matrix on PCP recovery is not possible due to the high concentrations of PCP in the original sample and the comparatively low levels of PCP added to the matrix spike samples. The water sample matrix spike sample analyzed by EPA Method 8270A resulted in high recoveries. These recoveries are on the high end of the QC acceptance criteria for PCP recoveries listed in EPA Method 8270A (14 to 176 percent recovery). However, the agreement between the matrix spike and matrix spike duplicate was excellent as determined by the RPD of the matrix spike recoveries. The water matrix spike samples analyzed using EPA Method 515.1 were affected by the concentration of PCP in the original sample. Although the matrix spike recoveries for sample 101 were found to be acceptable, the recoveries of PCP spiked into the sample were affected by the much larger concentration of PCP in the original sample. Sample 111 also was affected by the concentration of PCP in the original sample. Results of both the matrix spike sample and the matrix spike duplicate sample were less than the result for the original sample, which may indicate a heterogeneity problem with the sample. The low levels of PCP added to this sample were not enough to obtain an accurate indication of matrix spike recovery.

The EPA Region 7 Laboratory SMO prepared blank spike samples for water samples analyzed by EPA Methods 8270A and 5 15.1. These samples were used to evaluate the accuracy of the laboratory. Blank spike samples were stored, extracted, and analyzed in the same manner as all other samples. The percent recoveries of the blank spike samples fell within the 14 to 176 percent QC acceptance criteria listed in EPA Method 8270A and the 67.6 to 192.4 percent acceptance criteria listed in EPA Method 515.1. The accuracy of the analysis of water samples using EPA Methods 8270A and 5 15.1 was found to be acceptable, based on the blank spike sample results.

Data Reporting

The data report PRC received from the EPA Region 7 Laboratory included a standard EPA Region 7 Analysis Request Report. Results were reported on a dry weight basis, as required in the methods. PRC obtained data on the loss-ondrying determination for each of the samples. The loss-on-drying values were used to convert the confirmatory laboratory data from a dry weight basis to a wet weight basis.

Results were reported by the confirmatory laboratory in micrograms per kilogram **(µg/kg)** for soil samples and micrograms per liter **(µg/L)** for water samples. Soil sample results were converted to milligrams per kilogram (mg/kg) so they could be compared to the results from the technology, which reported results for soil samples in milligrams per kilogram. The technology's results for water samples

TABLE 5-1. SOIL MATRIX SPIKE SAMPLE RESULTS FOR EPA METHODS 8270A AND 8151A

Sample No.	Amount Found in Original Sample (ppm)	Amount Added to Matrix Spike and Duplicate (ppm)	Amount Found in Matrix Spike (ppm)	Percent Recovery (%)	Amount Found in Matrix Spike Duplicate (ppm)	Duplicate Percent Recovery (%)	Relative Percent Difference %
036	40.0	1.90	66.0	1,370	51 .o	579	81
048	30,000	46.0	22,000	0	24,000	0	0
053	2.30	1.50	12.0	647	5.20	193	108
073	86.0	11.0	130	400	93.0	64	145
087	46.0	1.40	57.0	786	64.0	1,285	48
089	0.247	0.098	0.315	69	0.241	0	200
098	0.70	0.41	0.82	29	0.98	68	80

TABLE 5-2. WATER MATRIX SPIKE SAMPLE RESULTS FOR EPA METHODS 8270A AND 515.1

Sample No.	Amount Found in Original Sample (ppb)	Amount Added to Matrix Spike and Duplicate (ppb)	Amount Found in Matrix Spike (ppb)	Percent Recovery (%)	Amount Found in Matrix Spike Duplicate (ppb)	Duplicate Percent Recovery (%)	Relative Percent Difference (%)
101	4.14	0.446	4.46	72	4.24	22	106
104	50.0 u	200	348	174	353	177	2
111	1.85	0.398	1.55	0	1.64	0	0

were reported in micrograms per liter, so no conversion of the confirmatory laboratory data was needed.

Data Quality Assessment

Accuracy refers to the difference between the sample result and the true concentration of analyte in the sample. Bias, a measure of the departure from complete accuracy, can be caused by such processes as loss of analyte during the extraction process, interferences, and systematic contamination or carryover of an analyte from one sample to the next. Accuracy for the confirmatory laboratory was assessed by using PE samples. Four of the PE samples used for this demonstration were purchased from Environmental Resource Associates (ERA). Two of these PE samples were soil and two were water. These samples contained a known quantity of PCP. ERA supplied data sheets for each PE sample that included the true concentration and an acceptance range for the sample. The acceptance range was based on the 95 percent confidence interval taken from data generated by ERA and EPA interlaboratory studies. A third water PE sample was prepared by the PRC lead chemist to widen the range covered by the PE samples.

These samples were extracted and analyzed in the same manner as the other water and soil samples. The confirmatory laboratory did not know which samples were PE samples or the certified values and acceptance ranges. The true value concentration of soil PE sample 099 (the low-level sample) was 7.44 ppm, with an acceptance range of 1.1 to 13 ppm. The result reported by the confirmatory laboratory for this sample was 4.02 ppm, which was within the acceptance range. The percent recovery of this sample by the confirmatory laboratory was 54 percent. The true concentration of soil PE sample 100 (the high-level sample) was 101 ppm with an acceptance range of 15 to 177 ppm. The result reported for this sample by the confiiatory laboratory was 52.4 ppm, which was within the acceptance range. The percent recovery of this sample by the confirmatory laboratory was 52 percent.

The true value concentration of water PE sample 106 (the low-level sample) was 68.4 ppb with an acceptance range of 10 to 120 ppb. The result reported by the confirmatory laboratory for this sample was 10.3 ppb, which was within the acceptance range. The percent recovery of this sample by the confirmatory laboratory was 15 percent. The true concentration of

water PE sample 107 (the high-level sample) was 2,510 ppb with an acceptance range of 377 to 4,420 ppb. The result reported for this sample by the confirmatory laboratory was 2,050 ppb of PCP, which was within the acceptance range. The percent recovery of this sample by the confirmatory laboratory was 82 percent. The true value concentration of water PE sample 113 (the PE sample prepared by PRC) was 7.50 ppb. No acceptance range was statistically determined for this PE sample. Instead, PRC established a 30 to 170 percent window of acceptable values around the true value result of the low-level PE sample. This window is consistent with both the acceptance ranges of the PE samples prepared by ERA and the QC Acceptance Criteria for PCP recovery stated in EPA Method 8270A. The confirmatory laboratory result for this PE sample was within the acceptance range. Based on results for all of the PE samples, the accuracy of the confirmatory laboratory was acceptable.

Precision refers to the degree of mutual agreement among individual measurements and provides an estimate of random error. Precision for results obtained by confirmatory laboratory was determined by using field duplicate samples. Normally, laboratory duplicates are used to determine precision. However, no laboratory duplicates were analyzed by the confirmatory laboratories. Field duplicates are two samples collected together but delivered to the laboratory with separate sample numbers. Typically, field duplicate samples are used to measure both sampling and analysis error. PRC established control limits for field duplicate RPDs. These control limits are similar to those used to determine matrix spike recovery acceptance control limits. To establish the control limits, all sample pairs that did not produce two positive results were removed from the data set. Then the RPD for each pair was calculated, and the mean RPD and standard deviation were determined. The lower control limit was set at zero because this would mean that the results from a duplicate and its sample matched perfectly. The upper control limit was set by multiplying the standard deviation by two and adding it to the mean RPD. The RPD of each sample pair was then compared to these control limits. Each sample pair RPD was expected to fall within the control limits.

Fourteen soil field duplicate samples were collected and analyzed by the confiiatory laboratory during this demonstration. Field duplicate samples represented 17 percent of all soil samples collected and analyzed. The original results ranged from 0.10 to 26,100 ppm. Duplicate sample results ranged from 0.09 to 30,260 ppm. RPD values for the soil field duplicate pairs ranged from 1 to 168 RPD. The mean RPD value of the soil field duplicate pairs was 33 percent, with a standard deviation of 47 percent. For the soil field duplicate pairs the control limits were found to be 0 to 128 RPD. Thirteen of the fourteen, or 93 percent, of the field duplicate sample pairs fell within this range.

Six water field duplicate samples were collected and analyzed by the confirmatory laboratory during this demonstration. Field duplicate samples represented 32 percent of all water samples collected and analyzed. The original results ranged from 0.175 to 1,810 ppb. The field duplicate sample results ranged from 0.63 to 2,020 ppb. RPD values for the water field duplicate pairs ranged from 0 to 113 RPD. The mean RPD value of the water field duplicate pairs was 30 percent with a standard deviation of 41 percent. For the water field duplicate pairs, the control limits were found to be 0 to 112 RPD. Five of the six, or 83 percent of the field duplicate sample pairs, fell within this range.

Completeness refers to the amount of data collected from a measurement process compared to the amount expected (Stanley and Vemer 1983). For this demonstration, completeness referred to the proportion of valid, acceptable data generated by the confirmatory laboratory. The completeness objective for this project was 95 percent. This demonstration resulted in the analysis of 98 soil samples, 14 soil sample duplicates, 2 soil PE samples, 10 water samples, 6 water sample duplicates, and 3 water PE samples. Results were obtained for all of these samples. Completeness for the confirmatory laboratory was 100 percent.

Confirmatory Laboratory Costs and Turnaround Times

The cost for performing PCP analysis by EPA-approved analytical methods varies from laboratory to laboratory. The cost of analysis depends on the number of samples submitted for analysis, the matrix, and the level of QC performed. The following costs are given as general guidelines. EPA Method 8270A analysis costs range from $250 to $400 per sample. EPA Method 8151A analysis costs range from $150 to $250 per sample. EPA Method 515.1 analysis costs range from $125 to $200 per sample. Turnaround times for samples submitted for analysis with EPA-approved analytical methods range from 14 to 30 days. The turnaround time also depends on the number of samples submitted for analysis, the matrix, and the level of QC performed. Faster turnaround times may be available for an additional cost.

Section 6
EPA Region 7 Supetfund Program: FASP PCP Method

The FASP PCP Method was developed by the EPA Region 7 Superfund Branch under the Field Investigation Team Contract for use at Superfund sites. It was designed to provide quick, accurate results to determine PCP concentrations in soil and water samples. This method can also detect and quantify other phenols. PCP concentrations are reported in either ppb or ppm for soils and in ppb for water. The method uses a GC equipped with a megabore capillary column and an FID and ECD to identify and quantify PCP. Gas chromatography is an EPA-approved method for determining PCP concentrations in soil, water, and waste samples. The FASP PCP Method is an abbreviated, modified version of the EPA-approved methods.

Operational Characteristics

The instruments and equipment required for the FASP PCP Method are not very portable because of their large sizes. The equipment used includes the following: (1) a Shimadzu GC-14A equipped with an FID and ECD, (2) a Shiiadzu AOC-14 autosampler, (3) a Shimadzu CR-4AX data system, and (4) a COMPAQ SLT/286 laptop computer. The GC weighs 100 pounds with its autosampler attached and measures 18 inches by 24 inches by 12 inches. The data system weighs another 25 pounds and is the same size. In addition to this equipment, the following laboratory equipment is necessary: (1) Nitrogen carrier gas; (2) a DB-5, 30-meter, 0.53~millimeter inside diameter, or equivalent capillary column; (3) l0-, 50-, and l00-microliter micropipettes; (4) l0-, 25-, 50-, l00-, and 5OO-microliter micro syringes; (5) l-, 5-, and lo-milliliter glass volumetric pipets; (6) 2- to lo-milliliter repipetter with Teflon liners; (7) l0-, 50-, and l00-milliliter glass volumetric flasks with ground-glass stoppers; (8) 2-milliliter glass vials withTeflon-lined cap for storing stock standards; (9) 2-milliliter autosampler vials with Teflon-lined cap; (10) a bubble flow meter used to check GC column flows; (11) 4-fluid-ounce standard bottles with Teflon-lined screw cap for calibration standard storage; (12) 5.75- and 9-inch disposable glass Pasteur pipets; (13) 40-milliliter extraction vials with Teflon-lined screw caps; (14) 13- by l00-milliieter test tubes with Teflon-lined screw caps; (15) stainless-steel spatulas; (16) top loader balance with O.Ol-gram accuracy; (17) a high speed vortex mixer; (18) a digital timer; (19) large and small pipet bulbs to fit volumetric and Pasteur pipets; (20) labels; (21) permanent markers; (22) paper towels; (23) surgical gloves; (24) safety glasses; (25) laboratory.coats or other protective clothing; (26) refrigerator; (27) a fume hood; and (28) floppy disks. The following reagents are needed for sample extraction and analysis using the FASP method: (1) pesticide-grade or equivalent methyl-tert-butyl ether; (2) granular, pesticide-grade sodium sulfate; (3) concentrated Fisher Scientific or equivalent sulfuric acid; (4) powdered sodium bicarbonate for neutralizing acid spills; (5) Fisher Scientific or equivalent potassium hydroxide; and (6) 98 percent purity or greater phenol standards.

The FASP PCP Method must be performed indoors in a temperature-controlled environment to protect the analytical equipment from moisture and temperature extremes. Most of the other equipment and reagents also require this protection. During this demonstration, the FASP method was performed in an air conditioned trailer.

Another logistical requirement of the technology is electricity. Electricity was provided to the trailer through a temporary power pole. An alternative source of electricity may be used, such as a gasoline-powered generator. The use of a generator allows the analytical equipment to be operated even at the most remotely located sites. Electricity is also required for various support equipment. The method requires using a fume hood to evacuate solvent and acid fumes from the work area. The fume hood may be vented directly outdoors or internally through a charcoal filter that traps harmful fumes. A refrigerator is needed to store the PCP standards. The refrigerator used for this demonstration was 3.5 cubic feet in size. Carrier, make-up, and FID flame gases are required for gas chromatography.

The work space required for the analytical equipment is 12 square feet. Another 8 square feet is needed to perform sample extraction and preparation steps. Storage space for equipment, reagents, and glassware also is needed. A solvent storage cabinet for flammable solvents is recommended for safely storing solvents. Soil and water samples should be cooled until sample analysis. They also should not be stored with PCP standards,

The operator chosen to analyze samples for this demonstration was Mr. Ramarao Rayavarapu, an employee of PRC. Mr. Rayavarapu earned a B.S. degree in civil engineering in 1983 from Andhra University in India. Mr. Rayavarapu also earned an M. S. in environmental engineering in 1990 from the University of Missouri. While at PRC, Mr. Rayavarapu has conducted RCRA facility inspections, compliance evaluation inspections, and field sampling and oversight work. His experience also includes using several types of wet chemical analytical methods. While attending the University of Missouri, Mr. Rayavarapu worked as a laboratory research assistant for 2 years, analyzing liquid and soil samples using a GC and a high performance liquid chromatograph. Mr. Rayavarapu was also the operator for a similar demonstration of the FASP Polychlorinated Biphenyl Method, also a GC method. Mr. Rayavarapu was trained for this demonstration by the lead chemist. Training included 1 week of hands-on work using and maintaining the GC, as well as training in specific procedures required for the extraction, preparation, and analysis of samples.

The FASP PCP Method involves two steps: sample preparation and sample analysis. Soil sample preparation involved weighing a known amount of soil into an extraction vial. A measured portion of sodium sulfate was added to and mixed with the soil in the extraction vial, A measured amount of methyl-tert-butyl ether was added to the extraction vial. The vial was then mixed with a vortex shaker for 2 minutes. The organic sample extract was now ready to be analyzed for PCP. In soil samples suspected of containing petroleum hydrocarbons, an acid-base cleanup was used to separate the petroleum hydrocarbons from PCP. The cleanup was performed by transferring an aliquot of the organic sample extract to a test tube. A small amount of a 37 percent solution of potassium hydroxide was added to the test tube and mixed with the organic sample extract for 1 minute. PCP in the organic sample extract was converted to a potassium salt that is soluble in the aqueous solution. After this basic wash, the organic solvent was removed from the test tube and the aqueous phase, containing the PCP-potassium salts, was washed twice with organic solvent. A small amount of a 1: 1 solution of sulfuric acid was carefully added to the test tube. The pH of the solution was lowered to less than 2 with the sulfuric acid solution. This step converted the PCP-potassium salts back to PCP, which was soluble in the organic solvent. The acidic aqueous extract was extracted three times with methyl-tert-butyl ether. The organic sample extract was adjusted to a known concentration and was ready for analysis.

Water extraction was performed by measuring a volume of water into an extraction vial. The pH of the sample was adjusted to greater than 10 with a 37 percent solution of potassium hydroxide. The basic water sample was extracted three times with methyl-tert-butyl ether, and the organic solvent containing petroleum hydrocarbons and other interferences was discarded. The basic water sample's pH was first adjusted to less than 2 with a 1: 1 solution of sulfuric acid and then extracted three times with methyl-tert-butyl ether. This organic sample extract contained PCP. The organic sample extract was concentrated to a known volume and was ready for analysis.

Sample preparation, as noted by the operator, was exhaustive and time-consuming and required an operator experienced in laboratory procedures. The sample preparation became more complicated when the sample had to undergo additional cleanup steps to remove matrix interferences. Sample analysis required that the operator be familiar with GC techniques. A minimum of 6 months experience in using a GC and a minimum of 1 month experience in analyzing PCP is recommended to effectively use the FASP PCP Method. The operator noted that operation of the GC was easier for those with prior GC experience and with a basic knowledge of analytical chemistry.

The ruggedness of field portable GC equipment has been demonstrated through years of use. Maintenance of the GC and other equipment is essential to ensure quick and accurate PCP results in the laboratory or field. Because of the rigors of working in the field, the equipment should be on site and operating correctly at least 1 to 2 days before beginning sample collection. This lead time allows any travel-induced damage to be corrected prior to sample collection. Routine maintenance includes gas bottle changes, septum changes, and column conditioning. Nonroutine maintenance may include column changes or electronic board replacement. Agreements with equipment suppliers for overnight delivery of replacement parts and in-the-field servicing by equipment service representatives may be required to provide all needed maintenance.

Instrument reliability was evaluated by monitoring the equipment's ability to maintain its calibration. An initial calibration (ICAL) was performed at the start of the demonstration. The instrument calibration was monitored daily through continuing calibrations (CCAL) using the mid-level concentration of the PCP standards. During the CCAL the peak response obtained is compared to the average peak response obtained during the ICAL. These responses are compared by determining the RPD between responses. An RPD value of less than or equal to 25 percent is required to continue sample analysis. If the RPD is higher than 25 percent, another ICAL must be performed. Over the 13 days of the demonstration, three ICALs and 13 CCALs were performed. Out of three ICALs, two were performed for soil analysis, and one was performed for water analysis. Eleven CCALs were performed for the analysis of soil samples, and two CCALs were performed for the analysis of water samples. CCALs were performed on each day of sample analysis. On the eleventh day of the demonstration, the CCAL for soil analysis was out of control as a result of analyzing soil samples from the Winona Post site, which contained large amounts of petroleum hydrocarbons. Due to the heavy contamination with petroleum hydrocarbons in the samples and extracts, the septa and the glass liner in the injection port became contaminated, and the column became saturated with hydrocarbons. After determining the problem, the septa and the glass liner were replaced, and the column was conditioned at elevated oven temperature to desorb the petroleum hydrocarbons. Another ICAL was performed after column conditioning; although the relative standard deviation (RSD) for PCP was 33 percent in the second ICAL, this RSD was deemed acceptable, and the average calibration factor was used for sample quantitation. The two CCALS performed after the second ICAL were within the new control limits.

A new ICAL was performed for the analysis of water samples. The CCAL performed for water analysis was within control limits; however, the CCAL performed at the end of the analysis did not fall within control limits. The retention time of the specific PCP peak also was monitored through the CCALs. The retention time of this peak shifted throughout the demonstration. To maintain an accurate retention time window for this peak, the window was adjusted.

Another reliability factor evaluated was the consistency of the data system to properly draw a baseline for standards and samples. Baselines were drawn consistently for samples from the former Koppers site. However, samples from Winona Post site were highly contaminated with petroleum hydrocarbons, causing the baseline to drift.

Operators of the method need to be aware of hazards due to the chemicals used, particularly methyl tert-butyl ether (MtBE), sodium sulfate, and sulfuric acid. MtBE is used to extract PCP from samples and to dilute standard solutions. MtBE is an explosive and flammable solvent and should be handled only under a flame hood. Care should be taken when using MtBE to avoid inhalation and direct contact with the liquid and fumes. Ignition sources such as open flames should also be avoided, and a dry chemical fire extinguisher should be available in case of fire. Sodium sulfate is used to chemically dewater soil samples. Sodium sulfate is a fine granular powder and can be a skin and eye irritant. Protective clothing and safety glasses should be worn when using sodium sulfate. Sulfuric acid is used to reduce the pH of the samples. Sulfuric acid can cause chemical burns when spilled on the skin. Chemical-resistant clothing, gloves, and safety glasses are required for protection from sulfuric acid. An adequate source of water should be available in case the acid comes in contact with the skin. Eyewash solutions also should be available.

This method uses various types of glassware to analyze samples. Using glass always presents a possibility of breakage and injury. Glassware should be handled in a safe, careful manner. Broken glassware should not be used and should be disposed of in a safe manner. The ECD contains Nickel-63, a radioactive material. The amount of radioactive material in the ECD is minimal, and it is stored in a sealed container. Nevertheless, the container must be checked for possible leakage twice a year. Leakage of the radioactive material above federally regulated limits: requires immediate attention. The FID uses hydrogen gas, which is explosive and stored under high pressure. Care must be taken to secure the hydrogen gas cylinders to avoid cylinder damage.

The total cost of the analytical equipment used during this demonstration was $23,214. This cost includes the GC equipped with an ECD and FID, the autosampler, the data system, all equipment required to set up the GC, and installation. Costs per component are: $13,149 for the GC; $3,865 for the autosampler; and $6,200 for the data system. Analytical equipment can also be rented from a number of companies. A general survey of current market costs for comparable equipment indicates ranges from $1,500 to $2,500 per month. Most rental companies require a minimum rental time period, but this may be negotiable. Rental rates usually include delivery and setup but do not include GC columns. Many companies also offer rent-to-own or lease-to-own options for analytical equipment. The cost of reagents and equipment needed to perform the extraction, preparation, and analysis of soil samples

using the FASP PCP Method is estimated to be $5,000. This demonstration required about 370 sample extractions and injections; these 370 sample extractions include initial sample analysis, subsequent sample dilutions, and QA/QC samples. At $5,000, this equates to about $13.50 per sample.

Operator costs will vary depending on the technical level of the operator. As discussed earlier, a minimum of 6 months experience in using a GC and a minimum of 1 month experience in analyzing samples for PCP is recommended. Other costs associated with this method include electricity, use of a refrigerator, use of an indoor work area, and disposal costs. This demonstration generated a 55-gallon drum of laboratory waste. The appropriate way to dispose of this waste is through an approved incinerator facility, with an estimated cost of $2,080 per drum of waste.

Performance Factors

The sensitivity of the FASP PCP Method depends on the detection limits of the FID and ECD. The sensitivity of the detectors, especially the ECD, is such that it always gives some background response; this response is called "noise level." For most methods that use gas chromatography, the detection limit is defined as the minimum amount of a compound that will give a response greater than three times the noise level of the instrument. The response of the low PCP standard was well above three times the noise level for the instrument.

The FASP PCP Method's detection limit depends on the type of detector used with the instrument. The FID has a lower sensitivity than the ECD and hence, the detection limits for the FID are higher than those for the ECD. The detection limit for soil samples from the former Koppers site was 0.8 ppm; for soil samples from the Winona Post site was 1.6 ppm. The detection limit for water samples, if analyzed using the FID was 0.2 ppm. The ECD was used to analyze water samples when the FID analysis produced a nondetect. The detection limit for the ECD was 0.5 ppb PCP. Detection limits were based on the low PCP calibration standard used, and the dilution or concentration factors produced during sample extraction and preparation.

The majority of the soil samples were silty clay to silty sand. Sodium sulfate was added to all soil samples to bind the moisture present. Some of the samples from the Winona Post site contained wood chips. The wood chips were not segregated before sample extraction. The operator noted that these matrix characteristics did not pose a physical problem with the extraction. Samples from the Winona Post site showed large interferences from petroleum hydrocarbons. The FASP PCP Method includes a cleanup step to eliminate contamination from petroleum hydrocarbons. However, even after extensive cleanup of the sample extract, the interferences from petroleum hydrocarbons were not totally eliminated. The interference from these petroleum hydrocarbons made the PCP quantitation very difficult. In addition, as evidenced by the matrix spike recoveries, PCP appeared to have been lost during these cleanup steps.

Some water samples from the Winona Post site contained an oil sheen on top of the water layer. The PCP carrier used at this site was diesel fuel. To reduce any matrix effects from free-phase petroleum, the pH of the water samples was increased to greater than 12. The water sample was then washed one time with MtBE to remove petroleum hydrocarbons. The MtBE wash was discarded. The water sample was then acidified to a pH less than 2.0. The acidified water sample was extracted with MtBE, and the MtBE extract was analyzed. This cleanup procedure worked well except for two samples. Samples 102 and 103, and their duplicates, showed large interferences from petroleum hydrocarbons even after the cleanup steps for petroleum hydrocarbons. These interferences may be due to inadequate washing of the water sample with MtBE prior to acidification and extraction. Additional cleanup may have eliminated this interference.

Another sample matrix effect was the high levels of PCP found in many samples. If the initial extract was analyzed and it produced a detect outside the method calibration range, the extract was diluted. Of 134 soil samples, 34 samples were diluted more than 10 fold. Any time dilution is attempted, measurement errors may be introduced.

The FASP PCP Method can use an autosampler to increase analysis efficiency. The autosampler allows samples to be analyzed 24 hours a day. Once loaded with samples, the autosampler can be operated without the presence of an operator. The FASP PCP Method's operator observed that up to 20 samples could be analyzed in each 24-hour period. More than 181 samples were weighed, extracted, cleaned, concentrated, and analyzed during the 13-day demonstration period, for an average of 14 samples per day. The sample throughput was reduced by the time-consuming cleanup process required for the samples from the Winona Post site.

The linear range of the analysis for PCP was established during the ICAL by analyzing the three different calibrators. The three concentrations used during the ICAL ranged from 4 to 400 ppb for the FID. These concentrations correspond to a PCP calibration range of 0.8 to 80 ppm in the soil samples. For water

samples, the calibration range for the FID was 0.2 to 20 ppm. The PCP calibration range for the ECD was 1 to 50 ppb. Any soil sample that exceeded a PCP concentration of 80 ppm, and any water sample that exceeded 20 ppb of PCP was diluted to bring the response within the calibration range. Forty-eight samples collected during this demonstration required dilution to obtain PCP peaks within the linear range of the calibration. Twenty-three of the samples required a 1: 10 dilution; 12 required a 1: 100 dilution; 11 required a 1: 1,000 dilution; and two required a 1: 10,000 dilution. The linear range for the FASP PCP Method is comparable to the linear ranges used in formal laboratory analysis.

Drift is a measurement of an instrument's variability in quantitating a known amount of a standard. The drift associated with the FASP PCP Method was evaluated through daily CCALs. The responses exhibited during the CCALs were compared to those exhibited during the ICAL. During sample analysis, the retention time of the PCP peak was observed to be shifting. The shifts were small, but they eventually exceeded the acceptable retention time ranges calculated during the ICAL. The corrective action for this drift involved establishing new retention time ranges. The retention times of the PCP peak for the CCALs were used to establish the new ranges.

Specificity

Sample interferences may arise due to the presence of soil organic matter, cocontaminants such as phthalates, other chlorophenols, and PCP carrier solvents. Interferences due to PCP carrier solvents are especially important because they have a strong potential to give false positive results.

Phthalates, which are common laboratory contaminants, are found in many of the plastic materials that can be used in the extraction process. Organic solvents used to extract PCP will also extract phthalates from the containers. To prevent such extraction of phthalates along with PCP, use of plastic materials in any stage of sample storage, extraction, or analysis is discouraged. Interferences may also be introduced by other means, such as carrier gas contamination and carryover from highly contaminated samples. Potential for carrier gas contamination is reduced by using ultra-high purity grade gases. The use of carrier gas filters also helps eliminate these problems. The extraction and analysis of highly contaminated samples followed by the extraction and analysis of less contaminated samples frequently results in sample carryover contamination. During this demonstration, sample carryover contamination was reduced by using disposable glassware. Glassware was replaced after the extraction and analysis of each sample. To reduce contamination of the GC and ECD, only those extracts that the FID indicated contained no PCP or too little to be detected were analyzed on the ECD. The use of solvent washes added to the GC after the analysis of highly contaminated samples also reduced sample carryover contamination.

Sample matrix interferences are more difficult to eliminate. Common manmade sample matrix interferants include phthalates, halogenated solvents, halogenated pesticides, polar halogenated compounds, and chlorinated paraffins. In addition, natural organics present in soil may affect PCP extraction. Research has shown that PCP not only adsorbs to soil organic matter but also forms complexes with humic material. The FASP PCP Method is based on the belief that adding acid to bring down the pH to less than or equal to 2.0 will free PCP from such complexes. A concern raised by EPA Region 7 was that PCP and pentachlorophenate elute at different times, resulting in incorrect identification and quantitation. Both of these compounds can be present at wood treatment facilities. Acidification of the soil sample should convert all of the pentachlorophenate ions into PCP.

Cross-reactivity from other phenols was not of concern, because gas chromatography can identify specific phenols. However, one problem associated with high concentrations of other phenols is that such a sample would require dilution, which would raise the detection limits for PCP. None of the samples analyzed during this demonstration contained large amounts of other phenols.

The major problem associated with PCP quantitation during this demonstration was interference caused by the carrier solvent used for PCP application. At the former Koppers site, PCP was applied to the wood by dissolving it into isopropyl ether and butane. At the Winona Post site, diesel fuel was the carrier solvent for PCP application. The samples from the former Koppers site did not show any carrier effect. PRC believes this was due in part to the high volatility of isopropyl ether and butane. Also, because these carriers elute well before PCP, they do not cause interference. However, sample quantitation was affected by the presence of petroleum hydrocarbons in the Winona Post samples, because PCP elutes within the petroleum hydrocarbon pattern. Although the FASP method includes cleanup steps to eliminate these interferences, interferences were not removed completely.

To evaluate the method for cross-reactivity, the lead chemist prepared 21 spiked samples. SS-01 through SS-17 were spiked soil samples, and SS-18 through SS-21 were spiked water samples. Samples SS-01 to SS-04 were spiked with 2,4,6-trichlorophenol; SS-05 to SS-08 were spiked with diesel fuel; SS-10 to SS-13 were spiked with 2,4-dichlorophenol; SS-14 to SS-17 were spiked with 2,3,4,6-tetrachlorophenol; and SS-09 was a blank sample. Water samples SS-18 to SS-21 were spiked with PCP and diesel fuel. All chlorophenols in soil samples were spiked at the 5 ppm level; the diesel fuel was spiked at a 50 ppm level in soil samples; and the PCP and diesel fuel were spiked in water samples at 50 ppb and 125,000 ppb, respectively.

The FASP PCP Method operator did not identify PCP in any of the samples spiked with chlorophenols. Samples spiked with diesel fuel did give false positive results for PCP. The mean PCP concentration in these samples was 2.85 ppm, which indicates that about six percent of the diesel fuel chromatograph was identified as PCP. The mean percent recovery from the samples spiked with 2,4,6-trichlorophenol was 75 percent. Similarly, the mean percent recoveries for 2,4dichlorophenol and 2,3,4,6-tetrachlorophenol were 76.8 and 68.8, respectively. This data shows that the method is capable of separating the phenols. The resultant recoveries are within control limits. The percent PCP recoveries in water samples ranged from 220 to 288 percent. The mean recovery of PCP was 245 percent, and the standard deviation was 30 percent. The diesel fuel interference is not thought to be the source of these high recoveries. These samples were analyzed by ECD, which is specific to halogenated compounds and should not detect petroleum hydrocarbons. Currently PRC has not identified the cause of these elevated recoveries.

Intramethod Assessment

Intramethod measures of the technology's performance included its results on reagent blanks, the completeness of its results, its intramethod accuracy, and its intramethod precision. Reagent blank samples were prepared by taking reagents through all extraction, cleanup, and injection steps of the analysis. Reagent blanks were prepared with each batch of 20 samples. Ten reagent blanks were prepared and analyzed during the demonstration: nine reagent blanks for soil analysis and one reagent blank for water analysis. No PCP was detected in any of the blanks above method detection limits. These results indicate that no laboratory-induced PCP contamination was present.

None of the FASP PCP Method data was invalidated, and results were reported for every sample analyzed. Therefore, the completeness was 100 percent for soil samples and 100 percent for water samples.

The surrogate standard used for the FASP PCP Method is 2,4,6-tribromophenol, which like PCP, is a polar compound. The partitioning of this compound during the extraction and cleanup process is similar to the partitioning of PCP. The surrogate was added to all blanks, samples, their duplicates, matrix spikes, and matrix spike duplicates at a level of 2 ppm. Surrogate recoveries were calculated only from the concentrated sample extract. Whenever the sample extract was not concentrated or the sample extract was diluted, no surrogate recoveries were reported. These samples were coded DL to indicate that the sample was diluted.

In all, 76 surrogate recovery values were obtained with an average surrogate recovery of 65.8 percent. The standard deviation of the surrogate recovery was 29.2 percent. Control limits for surrogate standards were defined as $\pm$ 3 standard deviations from the mean. For the surrogate standards analyzed during this demonstration, the calculated control limits were from 0 to 153 percent recovery. Two of the 76 surrogate standard recoveries were outside of these control limits. No corrective action was taken. These control limits are advisory limits only. Water samples were also spiked with 2,4,6-tribromophenol, at a concentration of 1 ppb. The surrogate recoveries were calculated from data generated by the FID. The surrogate recoveries from those samples were diluted and are flagged as DL.

Surrogate recoveries were not calculated from the ECD data because the surrogate showed a very high response on the ECD. This response exceeded the calibration limits of the instrument. In all, 15 surrogate recovery values were obtained. The average surrogate recovery was 30.8 percent and the standard deviation was 13.1. The surrogate recoveries ranged from 0 to 70 percent. Surrogate recoveries of all samples fell within the established control limits.

Intramethod accuracy was assessed for the FASP PCP Method by using PE samples and matrix spike and matrix spike duplicate samples. Five PE samples were analyzed during the demonstration, two for the soil matrix and three for the water matrix. Four of them were purchased from ERA. Water PE sample 113 was prepared by PRC. These samples were extracted and analyzed in the same way as the other soil and water samples. The operator did not know that the samples were PE samples, nor did the operator know the true concentrations and acceptance ranges.

The true concentration of soil PE sample 100 (the high-level sample) was 101 ppm, with an acceptance

range of 15 to 177 ppm. The result reported for this sample by the FASP PCP Method was 94.2 ppm, which was within the acceptance range. The true value concentration of soil PE sample 099 (the low-level sample) was 7.44 ppm, with an acceptance range of 1.1 to 13 ppm. The result reported by the FASP PCP Method was 4.44 ppm, which also was within the acceptance range. The true concentration of water PE sample 107 (the high-level sample) was 2,510 ppb, with an acceptance range of 377 to 4,420 ppb. The result reported for this sample by the FASP PCP Method was 1,712 ppb of PCP, which was within the acceptance range. The true value concentration of water PE sample 106 (the low-level sample) was 68.4 ppb; with an acceptance range of 10 to 120 ppb. The result reported by the FASP PCP Method was 78.2 ppb, which was within the acceptance range. The true value concentration of water PE sample 113 (the sample prepared by PRC) was 7.5 ppb, with an acceptance range of 3.75 to 11.3 ppb. The result reported by the FASP PCP Method for this sample was 42 ppb, which was outside the acceptance range. Accuracy of the samples analyzed by the FASP PCP Method was found to be 100 percent for soil analysis and 66 percent for water analysis based on the results of the PE samples. However, the accuracy for the water analysis could not have been improved unless all three sample results fell within the PE sample accuracy ranges.

Matrix spike samples were used to evaluate the extraction and analysis efficiency of the technology. They also were used to determine accuracy. Matrix spike samples were prepared by adding a known quantity of PCP to a sample. Matrix spike samples were spiked with PCP at a level of 10.0 ppm in soil samples and 50 ppb in water samples. The spiked sample was also duplicated to produce a matrix spike duplicate sample. Recovery results of the matrix spike samples and their duplicates are listed in Tables 6-l and 6-2. Six matrix spike samples and matrix spike duplicate sample pairs were extracted and analyzed using the FASP PCP Method for soil analysis. The recoveries for these samples ranged from 0 to 89 percent. Matrix spike and matrix spike duplicate results for sample 058D indicated 0 percent recoveries. The original sample contained about 3.0 ppm of PCP, which is not high enough to affect any spike recoveries. These results were excluded as outliers, and the remaining matrix spike and matrix spike duplicate pair results were used to evaluate the technology. The average recovery for these samples was 52 percent. The standard deviation was 39 percent. Based on this data, control limits for matrix spike recovery samples ranged from 0 to 130 percent recovery. All matrix spike samples analyzed fell within these control limits. One matrix spike and one matrix spike duplicate sample were analyzed during the analysis of water samples. These samples were spiked with PCP at a level of 50 ppb. The sample had an original PCP concentration of 45.5 ppb. The spike recoveries from these two samples were 193 and 173 percent. No statistical evaluation of the data was performed due to the small size of the data set. The mean recovery calculated from these two sample recoveries is 183 percent.

For this demonstration, three types of intramethod precision data was generated: data from laboratory duplicate samples, data from field duplicate samples, and data from matrix spike duplicate samples. Laboratory duplicate samples consist of two analyses performed on a single sample delivered to a laboratory. Six laboratory duplicate soil samples and one laboratory duplicate water sample were analyzed using the FASP PCP Method. Only samples with detectable levels of PCP were used for laboratory duplicate analysis. The initial analyses of the duplicate soil samples ranged from 107 to 9,777 ppm, and the single water sample result was 5,236 ppb. When the analysis was duplicated, the results ranged from 107 to 4,323 ppm for soil analysis, and the water sample result was 6,303 ppb. The results for the soil and water laboratory duplicate samples are presented in Table 6-3 and Table 6-4, respectively. Field duplicate samples were also analyzed during this demonstration. Field duplicates consist of two samples collected together, but submitted to the laboratory with separate sample numbers. PRC collected 14 field duplicate soil samples and 10 field duplicate water samples during this demonstration. The results for the soil and water field duplicate samples are presented in Table 6-5 and Table 6-6, respectively.

Typically, field and laboratory duplicate samples are used to determine error induced by sample collection and analysis. Laboratory duplicates are compared to a window of acceptable values, and if any fall outside that window, corrective action is taken by the laboratory. Field duplicates are used to identify sampling analysis and matrix variability. To control the problems usually detected by laboratory duplicates, PRC used only one operator for each technology. It was assumed that any variance in that operator's laboratory techniques would be the same for each sample, and therefore, statistically insignificant. For field duplicates, PRC put each sample through a homogenization process designed to ensure that there was little difference between the PCP concentration in a sample and its duplicate. Only in a very few samples does the homogenization appear not to have been complete. Because the samples were homogenized and because only one operator was used, PRC used the laboratory and field duplicates together to determine the technology's precision. There are 20 duplicate pairs for soil analysis and 11 duplicate pairs

TABLE 6-1. SOIL MATRIX SPIKE SAMPLE RESULTS FOR THE FASP PCP METHOD

Sample No.	Amount Found In Original Sample (ppm)	Amount Added To Matrix Spike Sample (ppm)	Amount Found In Matrix Spike Sample (ppm)	Percent Recovery (%)
004	0.82	9.60	8.4	79
033	ND	10.1	9.02	89
046	0.95	11.5	10.8	86
058D	3.2	10.8	2.67	0
087D	1.78	10.0	6.26	45
089	ND	10.2	1.44	14

Sample No.	Amount Added To Matrix Spike Duplicate Sample (ppm)	Amount Found In Matrix Spike Duplicate Sample (ppm)	Percent Recovery (%)	Relative Percent Difference (%)
004	15.3	12.8	78	1.3
033	9.4	8.5	90	1.1
046	11.8	11.5	89	3.4
058D	9.62	2.04	0	0
087D	9.95	10.65	89	66
089	10.6	1.84	17	19

Note:

ND Not detected above soil quantitation limit of 0.80 ppm

TABLE 6-2. WATER MATRIX SPIKE SAMPLE RESULTS FOR THE FASP PCP METHOD

Sample No.	Amount Found In Original Sample (ppb)	Amount Added To Matrix Spike Sample (ppb)	Amount Found In Matrix Spike Sample (ppb)	Percent Recovery (%)
1lOD	45.5	50.0	142	193

Sample No.	Amount Added To Matrix Spike Duplicate Sample (ppb)	Amount Found In Matrix Spike Duplicate Sample (ppb)	Percent Recovery (%)	Relative Percent Difference (%)
	50.0	132	173	11

Note:

ND Not detected above soil quantitation limit of 0.80 ppm

TABLE 6-3. SOIL LABORATORY DUPLICATE SAMPLE RESULTS FOR THE FASP PCP METHOD

Sample No.	Original Sample Result (ppm)	Laboratory Duplicate Sample Result (ppm)	Relative Percent Difference (%)
015	649	339	63
036	40.7	58.9	37
049	501	212	81
059	9,777	4,323	77
072	1,345	1,826	30
084	107	107	0

TABLE 6-4. WATER LABORATORY DUPLICATE SAMPLE RESULTS FOR THE FASP PCP METHOD

Sample No.	Original Sample Result (ppb)	Laboratory Duplicate Sample Result (ppb)	Relative Percent Difference (%)
112	5,236	6,303	18

TABLE 6-5. SOIL FIELD DUPLICATE SAMPLE RESULTS FOR THE FASP PCP METHOD

Sample No.	Original Sample Result (ppm)	Field Duplicate Sample Result (ppm)	Relative Percent Difference (%)
001	2.40	2.96	21
011	115	107	7
020	ND	ND	NA
030	32	40	22
040	11.5	11.8	3
048	29,773	30,464	2
050	1.12	1.84	49
055	1,928	2,580	29
058	30.7	3.2	162
059	9,777	4,731	70
073	59.8	70.7	17
074	486	545	11
086	3.93	3.57	10
087	9.32	1.78	137

Notes:

ND Not detected above soil quantitation limit of 0.80 ppm.

NA Not applicable due to ND result.

TABLE 6-6. WATER FIELD DUPLICATE SAMPLE RESULTS FOR THE FASP PCP METHOD

Sample No.	Original Sample Result (ppb)	Field Duplicate Sample Result (ppb)	Relative Percent Difference (%)
101	74.5	60.4	21
102	43,804	53,439	20
103	55,900	68,900	21
104	11,307	14,106	22
105	95.1	70	30
108	120	81.3	38
109	89	46	64
110	45	45.5	1
111	11.2	48.7	125
112	5,236	6,288	18

for water analysis. Intramethod precision was evaluated for each sample matrix using all the duplicate pairs.

Even the best technology that determines results quantitatively cannot reproduce its results every time. Therefore, PRC established control limits like those sometimes used to evaluate laboratory duplicates. These control limits were then used to determine whether the difference between a result from a duplicate and the result from its respective sample was reasonable. To establish the control limits, all sample pairs that did not produce two positive results were removed from the data population. The RPD for each sample pair was then calculated, and the mean RPD and population standard deviation were The lower control limit was set at 0, which means that the results from a. duplicate

and its sample matched perfectly. The upper control limit was set by multiplying the standard deviation by two and adding it to the mean RPD. The RPD of each sample pair was then compared to these control limits. Each was expected to fall within them.

The FASP PCP Method detected PCP in both the sample and its duplicate in 19 of the 20 sample pairs for soil analysis and in all duplicate pairs for water analysis. Control limits were established for each sample matrix. For the soil matrix, the mean RPD was calculated to be 43.6, with a standard deviation of 45.5. Therefore, the control limits were set at 0 and 135. Two of the sample duplicate RPDs fell outside the range. The resulting precision is 89 percent, which is less than the required 90 percent, and thus unacceptable. Similarly, the control limits for RPDs established for water sample duplicates ranged from 0 to 102 percent. One RPD was outside control limits (sample pair 111). This data shows that 90 percent of the sample RPDs fell within the control limits, so the precision of the technology was deemed acceptable for water samples.

Matrix spike duplicate samples were used to further evaluate the precision of this method. RPD values for the six sets of soil matrix spike and matrix spike duplicate samples ranged from 0 to 66 percent. The mean RPD for these samples was 15 percent, and the standard deviation was 26 percent. The resultant upper control limit was 67 percent. All RPD values for the matrix spike and matrix spike duplicates fell within this range. Only one matrix spike and matrix spike duplicate sample was analyzed for water. Based on the small size of the data set, no control limits were calculated. However, the RPD value for this sample was 11 percent.

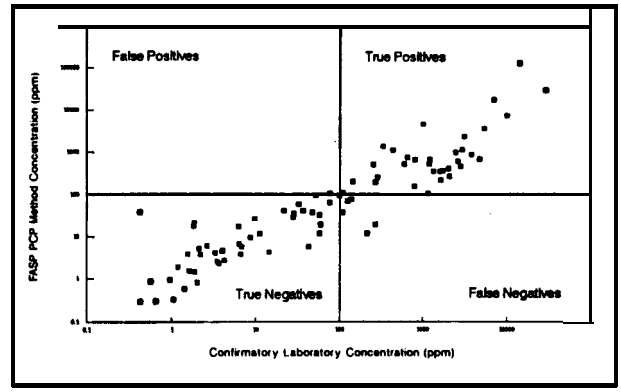
Comparison of Results to Confirmatory Laboratory Results

PRC used the statistical methods detailed in Section 3 to determine how well the results from the FASP PCP Method matched those from me confirmatory laboratory. The results used for the various data sets are presented in Tables 6-7 through 6-9, and a summary of the statistics is presented in Table 6-10. The purpose of this statistical data evaluation is to assess whether the method meets Level 3 criteria for accuracy and precision.

Soil Samples: Intermethod Accuracy

The initial linear regression analysis on the entire data set was based on results from 84 samples. The other results indicated that no PCP was detected above the method's detection limits. Figure 6-l illustrates the comparability between the FASP PCP Method and confirmatory data. A hypothetical 100 ppm action level

FIGURE 6-1 TOTAL SOIL DATA SET.



is also noted on this figure. The r^2 for this regression was 0.35, indicating that little or no relationship exists between the data sets. However, a residual analysis of the data identified samples 21, 44, 47, 48, 55, 59, 60, 75, and 76 as outliers. PRC removed these nine points and recalculated the linear regression. This regression had an r^2 of 0.41, indicating that there is still little correlation between the two data sets. The Wilcoxon Signed Ranks Test indicated that the FASP PCP Method's data was significantly different from that of the confirmatory laboratory. This supported the regression analysis. These results indicate that this technology is not accurate and that it cannot be mathematically corrected to estimate corresponding confiiatory data. Based on these results and depending on the intended data use, all of the samples analyzed by this method may need confirmation analysis. This places this technology, for the combined soil data set, into the Level 1 data quality category.

The second tier of the accuracy evaluation involved the separation of the data by site. This tier of data evaluation was conducted to assess potential carrier effects on the method's performance. Fifty samples from the former Koppers site were initially used for the regression analysis. Figure 6-2 illustrates the comparability between the FASP PCP Method and confirmatory data. The r^2 for this regression was 0.37, indicating that a relationship may exist between the data sets. A residual analysis of the data identified samples 21, 44, 47 and 48 as outliers. PRC removed these four points and recalculated the linear regression. When the regression was recalculated on the 46 remaining sample results, it defined an r^2 factor of 0.82, indicating that little or no relationship exists between the two data sets. The Wilcoxon Signed Ranks Test also indicated, at a 90 percent confidence level, that the FASP PCP Method's data was not significantly different from that of the confirmatory laboratory. Based on these results, 10 to 20 percent of the samples

FIGURE 6-2 FORMER KOPPERS SITE SOIL SAMPLES.

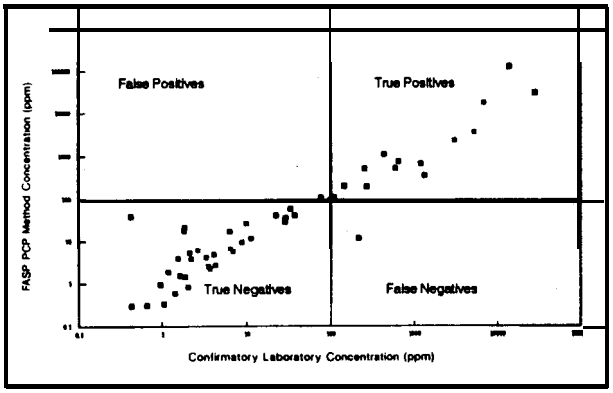
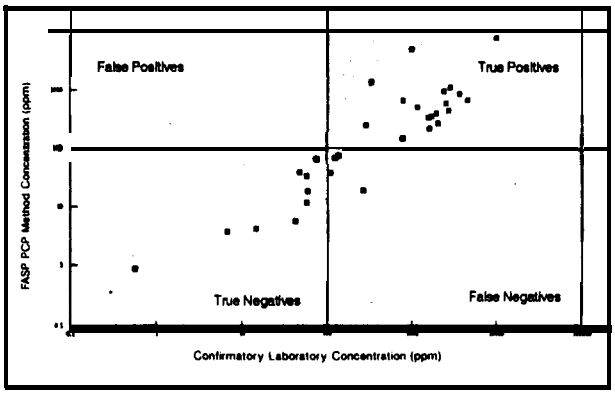


FIGURE 6-3 WINONA POST SOIL SAMPLES.



analyzed by this method may need confirmation analysis. This places this technology, for the combined soil data set, into the Level 1 data quality category.

The second tier of the accuracy evaluation involved the separation of the data by site. This tier of data evaluation was conducted to assess potential carrier effects on the method's performance. Fifty samples from the former Koppers site were initially used for the regression analysis. Figure 6-2 illustrates the comparability between the FASP PCP Method and confirmatory data. The r^2 for this regression was 0.37, indicating that a relationship may exist between the data sets. A residual analysis of the data identified samples 21, 44, 47 and 48 as outliers. PRC removed these four points and recalculated the linear regression. When the regression was recalculated on the 46 remaining sample results, it defined an r^2 factor of 0.82, indicating that little or no relationship exists between the two data sets. The Wilcoxon Signed Ranks Test also indicated, at a 90 percent confidence level, that the FASP PCP Method's data was not significantly different from that of the confirmatory laboratory. Based on these results, 10 to 20 percent of the samples analyzed by this method need confirmation analysis to calculate a correction factor, converting the FASP PCP method's data into estimates of corresponding confirmatory data. This places this method, for the Koppers soil data set, into the Level 2 data quality category.

Thirty-two samples from the Winona Post site were initially used for the second tier regression analysis. Figure 6-3 illustrates the comparability of the FASP PCP Method and confrrrnatory data. The r^2 for this regression was 0.53, indicating that a relationship may exist between the data sets. A residual analysis of the data identified samples 59, 60, 72, and 77 as outliers. PRC removed these four points and recalculated the linear regression. The regression then defined an r^2 factor of 0.71. This is slightly below the 0.75 required for Level 2 classification. The Wilcoxon Signed Ranks Test indicated that the FASP PCP Method's data was significantly different from that of the confirmatory laboratory, which supported the findings of the regression analysis. These results indicate that this technology is not accurate, and that it cannot be mathematically corrected to estimate corresponding confirmatory data. Depending on the data quality requirements, all samples analyzed by this technology may need confirmatory analysis. This factor places this technology, for the Winona Post soil data set, in the Level 1 data quality category.

The demonstration results indicate that there is a carrier effect on this method. The accuracy and comparability to the confiiatory data is higher in the samples contaminated with PCP in an isobutyl ether and butane carrier. The diesel fuel carrier solvent causes interference that lowers accuracy and comparability with confirmatory data.

The data was also evaluated by concentration. Overall, the confirmatory laboratory found that 46 samples had concentrations of less than 100 ppm. The initial linear regression of these 46 samples defined an r^2 of 0.63, indicating that a relationship may exist between the two data sets. However, a residual analysis of the data identified samples 14, 38, 85 and 100 as outliers. PRC removed these four points, recalculated the linear regression, and defined an r^2 of 0.50, also indicating that a relationship may exist between the two data sets. However, the Wilcoxon Signed Ranks Test indicated, at a 90 percent confidence level, that the FASP PCP Method's data was not significantly different from that of the confiiatory laboratory. This contradicts the regression analysis and indicates that one of the data sets may not have normal distribution. For this reason, the regression data was considered suspect. The results, therefore, indicate that this technology

TABLE 6-7. SUMMARY OF DEMONSTRATION DATA: FORMER KOPPERS SITE SOIL SAMPLES

Sample No.	FASP PCP Method (0.80 ppm)[a]	Confirmatory Laboratory (ppm)	Sample No.	FASP PCP Method (0.80 ppm)[a]	Confirmatory Laboratory (ppm)
001	2.40	4.42	027	11.5	11.30
001 D	2.96	4.18	028	<0.80 U	0.45
002	1.50	1.64	029	0.33 J	1.06
003	<0.80 U	0.13[b]	030	32.0	28.60
004	0.82	2.04	030D	40.0	29.00
005	2.24	3.70	031	0.58	1.43
006	1.44	1.89	032	17.2	0.62[b]
007	6.00 J	2.66	033	<0.80 U	0.40
008	0.31 J	0.66	034	<0.80 U	0.31[b]
009	2.50	3.52	035	201	145.0
010	1,100	435.0	036	40.7	36.80
011	115	106.0	037	1.86	1.19
011D	107	112.0	038	107	77.00
012	<0.80 U	0.056[b]	039	4.10	3.32
013	58.2	32.80	040	11.5	400.0
014	93.9	99.60	040D	11.8	34.40
015	649	1,190	041	6.53	6.44
016	195	273.0	042	4.72	4.09
017	346	1,335	043	739	655.0
018	5.20 J	2.13	044	17,600	6,956
019	5.70 J	6.89	045	41.0	22.10
020	<0.80 U	0.10	046	0.95	0.95
020D	<0.80 U	0.09[b]	047	127,000	13,920
021	3,600	5,320	048	29,800	26,100
022	17.6	1.85	048D	30,500	30,260
023	21.6	1.86	049	501	255.0
024	3.87	1.57	050	1.12J	2.16
025	521	593.0	050D	1.84	1.25
026	39.1	0.42	051	0.30 J	0.43
052	29.2	28.20	056	26.1	9.90
053	3.67	2.23	057	9.35	8.74
054	<0.80 U	0.47	058	30.7	3.53
055	1,930	3,135	058D	3.20	9.13
055D	2,580	3,003			

Notes:

a Detection limit.
D Sample analyzed by Method 8151 A; all other samples were analyzed by Method 8270A.
J Reported amount is below detection limit or not valid by approved QC procedures.
U PCP was not detected.

TABLE 6-8. SUMMARY OF DEMONSTRATION DATA: WINONA POST SITE SOIL SAMPLES

Sample No.	FASP PCP Method (1.60 ppm)[a]	Confirmatory Laboratory (ppm)	Sample No.	FASP PCP Method (1.60 ppm)[a]	Confirmatory Laboratory (ppm)
059	9,780	**9,600**	D80	605	2,550
059D	4,730	10,260	081	70.0	125.0
060	4,570	1,008	082	965	2,400
061	457	2,744	083	18.6	270.0
062	76.0	138.0	084	107	1,140
063	219	1,610	085	11.5	57.70
064	408	1,978	086	3.93	6.59
065	348	1,577	086D	3.57	6.88
066	32.4	57.80	087	9.32	34.00
067	37.4	110.0	087D	1.78	51.80
068	38.0	47.70	088	**<1.60** U	2.58
069	664	798.0	089	**<1**.60 U	0.21b
070	1,120	2,888	090	<I .60 U	**0.55**[b]
071	249	289.0	091	**<1.60** U	**0.28**[b]
072	1,350	336.0	092	0.87 J	**0.57**[b]
073	59.8	74.80	093	**<1.60** U	**0.19**[b]
073D	70.7	78.20	094	**<1.60** U	**1.02**[b]
074	486	836.0	095	**<1.60** U	**0.088**[b]
074D	545	1,520	096	18.2	59.80
075	860	3,692	097	4.16	14.60
076	679	4,590	098	>I .60 U	0.57
077	273	2,040	099	4.44	4.02
078	361	1,720	100	94.2	52.40
079	155	792.0			

Notes.

[a] Detection limit; this value was raised due to interference from the diesel fuel carrier.
[b] Sample analyzed by Method 8151A; all other samples were analyzed by Method 8270A
J Reported amount is below detected limit or not valid by approved QC procedures.
U PCP was not detected.

meets Level 2 criteria, when analyzing samples with concentrations of less than 100 ppm, but its data cannot be mathematically corrected to estimate corresponding confiitory data.

The confirmatory laboratory identified 38 samples with concentrations greater than 100 ppm. The initial linear regression on these 38 samples defined an r^2 of 0.31, indicating that little or no relationship exists between the two data sets. A residual analysis of the data identified samples 44,47,48, 59 and 60 as outliers. PRC removed these five points, recalculated the regression, and defined an r^2 of 0.41. The Wilcoxon Signed Ranks Test indicated that the FASP PCP Method's data was significantly different from that of the confirmatory laboratory, which confirmed the regression analysis. These results indicate that this technology is not accurate and that it cannot be mathematically corrected to estimate corresponding confiiatory data. This factor places this technology, for the combined soil data set for PCP concentrations above 100 ppm, in the Level 1 data quality category. The data indicates a

TABLE 6-9. SUMMARY OF DEMONSTRATION WATER DATA

Sample No.[a]	FASP PCP Method (0.002 ppm)[b]	Confirmatory Laboratory (ppm)
101	0.075	0.00414[c]
102	44	15.90
103	56	13.50
104	11	0.0123
105	0.095	0.849
105D	0.070	0.640
106	0.078	0.0103[c]
107	4.4	2.050'
108	0.12	0.00185[c]
108D	0.81	0.00221[c]
109	0.089	0.000175[c]
109D	0.046	0.00063[c]
110	0.045	0.0181[c]
110D	0.046	0.0181[c]
111	0.011 J	0.000348[c]
111D	0.49	0.00032[c]
112	5.2	1.810
112D	6.3	2.020
113	0.042	0.00227[c]

Notes:

a Samples 101 through 105D were from the VVinona Post site; Samples 108 through 112D were detected from the former Koppers site. Samples 106,107 and 113 were PE samples.

b Detection limit

c Sample analyzed by Method 515.1; all other samples were analyzed by Method 8270A.

J Reported amount is below detection limit or not valid by approved QC procedures.

TABLE 6-10. SUMMARY OF REGRESSION AND RESIDUAL STATISTICS SOIL ACCURACY

	N	r^2	Y-int	Slope	Wilcoxon Probability
All Data	75	0.41	70.1	0.26	Significant Difference
All Data <100 ppm	42	0.50	5.3	0.64	No Significant Difference
All Data >100 ppm	33	0.41	116.7	0.34	Significant Difference
Koppers-All Data	46	0.82	28.4	0.68	No Significant Difference
Koppers <100 ppm	31	0.76	2.0	1.2	Significant Difference
Koppers >100 ppm	13	0.71	-779.9	1.85	Significant Difference
Winona-All Data	28	0.71	27.4	0.24	Significant Difference
Winona <100 ppm	9	0.51	-4.64	0.61	Significant Difference
Winona >100 ppm	19	0.57	84.4	0.23	Significant Difference

Notes:

N Number of data points

r^2 Coefficient of determination adjusted for variance

Y-int Y-axis intercept of the regression line

trend for the technology to have slightly better accuracy and comparability when used to analyze samples containing less than 100 ppm PCP.

The confirmatory laboratory found that 35 samples from the former Koppers site had concentrations of less than 100 ppm. The initial linear regression on these samples defined an r^2 of 0.84, indicating that a relationship exists between the two data sets. A residual analysis of the data identified samples 13, 14, 26, and 38 as outliers. PRC removed these four points, recalculated the regression,and defined an r^2 of 0.76, indicating that a relationship exists between the two data sets. The Wilcoxon Signed Ranks Test indicated, at a 90 percent confidence level, that the FASP PCP Method's data was significantly different from that of the confirmatory laboratory. These results indicate that this technology is not accurate, but that it can be mathematically corrected to estimate correspo nding confirmatory data. Based on these results, 10 to 20 percent of the samples analyzed by this method need confirmation analysis. This factor places this technology, for these samples, in the Level 2 data quality category.

The evaluation of the Koppers data for those samples the confirmatory laboratory found had results greater than 100 ppm was based on 15 samples. The initial regression defined an r^2 of 0.27, indicating that

little or no relationship exists between the two data sets, but samples 47 and 48 were identified as outliers. PRC removed these two points, recalculated the regression, and defined an r^2 of 0.71 indicating that a relationship may exist between the two data sets. The Wilcoxon Signed Ranks Test confirmed the regression data, indicating that the FASP PCP Method's data was significantly different from that of the confirmatory laboratory. These results, therefore, indicate that this method is not accurate and cannot be reliably mathematically corrected to estimate corresponding confirmatory data. This places the method, for this data set, in the Level 1 data quality category. This data indicates that the method tended to have greater accuracy and comparability to confirmatory data for samples containing less than 100 ppm PCP.

The same types of analyses were conducted on data from samples from the Winona Post site. The confirmatory laboratory found that nine of the samples had concentrations less than 100 ppm. The initial regression defmed an r^2 of 0.51, indicating that a relationship may exist between the two data sets. A residual analysis of the data identified no outliers. The Wilcoxon Signed Ranks Test indicated that the FASP PCP Method's data was significantly different from that of the confirmatory laboratory. These results indicate that this technology is not accurate and that it cannot be mathematically corrected to estimate corresponding confirmatory data. This factor places this technology, for Winona Post site samples with less than 100 ppm PCP, in the Level 1 data quality category.

The confirmatory laboratory found that 23 samples from Winona Post site had concentrations greater than 100 ppm PCP. The initial regression defined an r^2 of 0.49, indicating that little or no relationship exists between the two data sets. A residual analysis of the data identified samples 26, 59, 60 and 80 as outliers. PRC removed these points, recalculated the regression, and defined an r^2 of 0.57, indicating that a relationship may exist between the two data sets. The Wilcoxon Signed Ranks Test verified these results, which indicates that this technology is not accurate and that it cannot be mathematically corrected to estimate corresponding confirmatory data. All samples analyzed by this method need confumation analysis. This factor places this technology, for these samples, in the Level 1 data quality category. This data indicates a tendency for this method to be more comparable to the confirmatory data for samples containing less than 100 ppm PCP. This conclusion is based on an examination of the slopes and y-intercepts for the data sets.

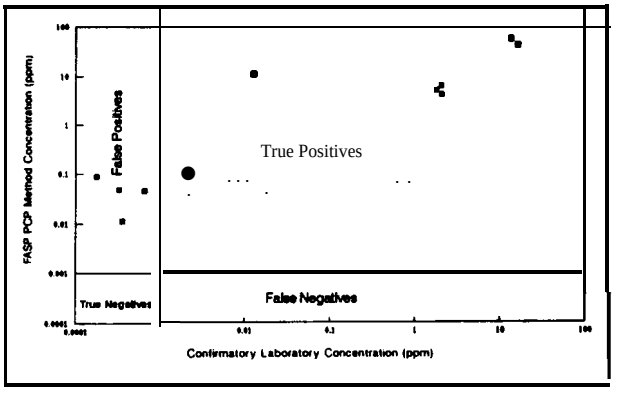
Soil Samples: Intermethod Precision

When the Dunnett's Test compared the RPDs between the FASP PCP Method's entire field duplicate data set and the corresponding confirmatory laboratory data set, the precisions were found to be statistically similar. When the precision was examined relative to the site from which the duplicates were collected, the precision between the methods were found to be statistically similar at both sites. The Wilcoxon Signed Rank Test confirmed this conclusion.

Water Samples: Intermethod Accuracy

Nineteen water samples were found to have concentrations of PCP above detection limits by both the confirmatory laboratory and FASP methods. These samples, therefore, were eligible for the regression analysis. Figure 6-4 illustrates the comparability between the FASP PCP Method and confirmatory data. The EPA maximum contaminant level (MCL) of 1 ppb is also shown on this figure. The r^2 for this regression was 0.92, indicating that a relationship exists between the data sets. The regression analysis produced a slope of 3.3, and a y-intercept of 0.36. However, residual analysis of this data identified samples 102 and 103 as significantly influencing the regression. When these points were removed as outliers, the r^2 dropped to 0.18 and the slope and intercept changed to 12.0 and 0.76 respectively. This confirmed the strong influence these points exhibited over the regression analysis. The Wilcoxon Signed Ranks Test was used to verify these results. It indicated, at a 90 percent confidence level, that the FASP PCP method's data was significantly different from that of the confirmatory laboratory. This supported the regression analysis. These results indicate that this technology is not accurate and that its data

FIGURE 6-4 TOTAL WATER DATA SET.



cannot be mathematically corrected to estimate corresponding confirmatory data. This places the method, for the combined water data set, in the Level 1 data quality category.

Of these samples, 10 were collected at the former Koppers site. The r^2 for this regression was 0.99, indicating that a strong relationship exists between the data sets. Figure 6-5 illustrates the comparability between the FASP PCP Method and confirmatory data. An examination of the slope (3.0) and y-intercept (0.042) showed that both of these parameters are not statistically equivalent to their expected values. The Wilcoxon Signed Ranks Test was used to verify these results, but it indicated that the FASP PCP Method's data was significantly different from that of the confirmatory laboratory. The method, therefore, does not produce Level 3 data, but rather Level 2 data. The data produced from this technology must be corrected to simulate confirmatory data by submitting 10 to 20 percent of the samples for confirmatory analysis.

Six of the 19 water samples eligible for regression analysis were from Winona Post site. The r^2 for regression on these six samples was 0.87, indicating that a strong relationship exists between the data sets. Figure 6-6 illustrates the comparability between the FASP PCP Method and confirmatory data. An examination of the slope (3.2) and y-intercept (2.2) showed that both of these parameters are not statistically equivalent to their expected values. A residual analysis of this data, though, showed that samples 102 and 103 had a large influence on the regression, and when these outliers were removed and the regression was recalculated, the r^2 was zero.

The Wilcoxon Signed Ranks Test, however, indicated at a 90 percent confidence level that the FASP PCP Method's data was not significantly different from that of the confirmatory laboratory, without the outliers. The regression analysis, therefore, did not agree with the Wilcoxon Signed Ranks test when it was run on the data set without the outliers. This indicates that the condition of normality was not satisfied by one or both of the data sets, and it makes the regression analysis suspect. This indicates that the technology does not produce Level 3 data but rather Level 2 data. Data produced from this technology is statistically similar to confirmatory data

FIGURE 6-5 FORMER KOPPERS SITE WATER SAMPLES.

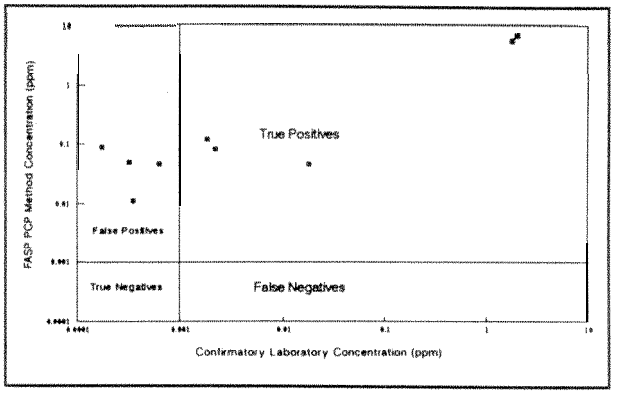
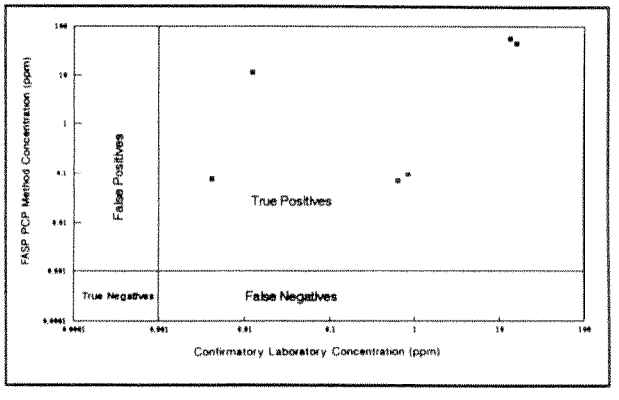


FIGURE 6-6 WINONA POST WATER SAMPLES.



but cannot be corrected. This data indicated that the method is affected by PCP carrier interferences. This method produced the greatest accuracy and comparability to confirmatory data for PCP in an isobutyl ether and butane carrier.

Wafer Samples: Intermethod Precision

For water sample analysis, the Dunnett's Test compared RPDs between the FASP PCP Method's field duplicate data set and the corresponding confirmatory laboratory data set. This indicates that the technology's precision is not different from the confirmatory laboratory's. The Wilcoxon Signed Rank Test confirmed this data.

Section 7
Applications Assessment

The principal advantage of the FASP PCP Method is that it is very specific to PCP. This specificity reduces the chances of determining that a sample contains PCP when, in fact, it does not. This specificity also greatly reduces the chances of determining that a sample contains no PCP, when it actually does. Other advantages of the FASP PCP Method include the following: (1) it is inexpensive when compared to formal laboratory analysis using EPA-approved methods for PCP, (2) it is portable enough to use in the field, (3) it has a high sample throughput, and (4) it is capable of providing sample results quickly. In addition, the detection limit for water samples analyzed with the FASP PCP Method is less than the MCL of 1.0 ppb. The MCL is an EPA-enforceable action limit for PCP in water samples.

The FASP PCP Method provides quantitative estimates of PCP concentrations in soil and water samples. It estimates sample results through a quantitative comparison to a standard curve. However, its estimation of PCP concentrations in samples may not always agree with results from the analysis of the same sample by EPA-approved methodologies. The results from this demonstration indicate that this technology has a carrier solvent sensitivity. The technology produced data that were more consistent with confirmatory results for samples from the former Koppers site. The high level of diesel fuel contamination in the Winona Post site samples required extensive cleanup steps to remove interferences. These cleanup steps were not sufficient to remove all of the interferences. Thus, the Winona Post site results were all significantly different than the confirmatory results. Method results, when compared to confirmatory laboratory results, may include both false positive results, which overestimate the concentration of PCP in the sample, and false negative results, which underestimate the concentration of PCP in the sample. Both false positive and false negative results have important implications on investigative and remedial activities. Another limitation of the FASP PCP Method is that it can be affected by chemicals found naturally in environmental samples, such as humic acids, as well as by chemicals associated with PCP treatment of wood products. These chemicals may affect the technology's performance even when cleanup steps are employed.

The FASP PCP Method is best operated by individuals with at least 6 months of experience using a GC and at least 1 month of experience performing PCP analysis. Logistical limitations of the FASP PCP Method include the need for a large capital investment for the purchase of the analytical instrumentation and extraction equipment. This equipment may also be rented. The FASP PCP Method's equipment requires electricity for operation, and the instruments must be operated indoors in a temperature-controlled environment. The method uses hazardous chemicals such as MtBE, sodium hydroxide, and sulfuric acid. Proper safety and disposal practices need to be employed when using the FASP PCP Method.

The FASP PCP Method is designed to provide quantitative screening results for PCP in water and soil samples. Applications for the FASP PCP Method include both laboratory and field uses. The FASP PCP Method can be used by laboratories as a rapid screening tool for PCP. Its results can be used to determine appropriate sample extraction techniques as well as to determine dilutions that may be required for sample analysis. Its results also can be used to determine the appropriate analytical method to be used for confirmatory sample analysis. The use of this method in this mode can protect more sensitive instruments from damage exposure resulting from highly contaminated samples. The FASP PCP Method also can be used to guide the following field investigations and sample collection activities: (1) determining the vertical and horizontal extent of PCP contamination in soil, (2) tracking PCP groundwater contamination plumes, and (3) determining PCP contamination in surface waters. Another use of the FASP PCP Method is to monitor the effectiveness of remediation techniques employed to reduce or eliminate PCP contamination. In

particular, it can be used to determine whether PCP concentrations in soil or water samples exceed site-specific action limits.

The FASP PCP Method is best used at sites where PCP is a known contaminant where petroleum products are not the carrier solvents, and where large concentrations of other organic chemicals are not present in the samples. Generally, the larger the site or the larger the number of samples collected, the greater the advantage of using the FASP PCP Method. The use of this method at large sites will decrease the cost of the investigation by decreasing the number of samples requiring confirmatory laboratory analysis and by enabling more work to be completed during a single sampling visit. The primary advantage of this method is that it can allow work to continue without having to wait for confirmatory laboratory results.

The technology can be used to guide field work and sampling efforts, provided that at least 10 to 20 percent of the samples are submitted to a confirmatory laboratory for EPA-approved method analysis. These samples must constitute PCP concentrations from all levels found. Results from the confirmatory laboratory can be used to formulate correction factors for the technology's results or verify its similarity to the confirmatory data. Correction factors, if needed, can be applied to its results to obtain a more precise estimation of PCP contamination. Correction factors are site specific and may, in fact, be sample-matrix specific. Any time sites or sample matrices change, new correction factors should be established by comparing the FASP PCP Method's results to confirmatory laboratory results of the same samples.

Particular attention needs to be paid to samples with PCP concentrations near the action limit of the site, because the method can provide both false positive results and false negative results when compared to confirmatory laboratory results. False positive results will cause remediation efforts to be performed in areas that do not require cleaunup False negative results will cause no remediation to take place in areas where cleanup is needed. To limit the impact of false negative results, working action levels for this method should be set at 80 to 90 percent of the target action level. Field investigators should recognize that the FASP PCP Method is designed as a screening tool to assist in evaluating PCP contamination. The technology is an abbreviation of an approved method for determining PCP concentrations. When in doubt, samples should be submitted for confirmatory laboratory analysis using EPA-approved methods.

Section 8
References

PRC Environmental Management, Inc. (PRC). 1993a. "Predemonstration Sampling Plan For The Evaluation of Pentachlorophenol Field Screening Technologies." EPA Contract 68-CO0047.

PRC. 1993b. "Final Demonstration Plan for the Evaluation of Pentachlorophenol Field Screening Technologies." EPA Contract No. 68-CO-0047.

Stanley, T. W. and S. S. Vemer. 1983. "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans." U.S. Environmental Protection Agency, Washington DC. EPA/600/4-83/004.

U.S. Enviromnental Protection Agency. 1990. "Quality Assurance/Quality Control Guidance for Removal Activities." EPA/540/G-90/004. April.

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